## Virginia Wood Preserving Site Richmond, Virginia

Work Plan and Quality Assurance Project Plan

Appendices E, F, and G

Prepared for:

Rentokil, Incorporated Supa Timber Division



AR300513

April 3, 1989

AR300514

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APPENDIX E

CompuChem Laboratories Quality Assurance Plan

Quality Assurance Plan

CompuChem® Laboratories

P. O. Box 12652

3308 Chapel Hill/Nelson Highway

Research Triangle Park

North Carolina 27709

This document conforms to "Guidelines and Specifications for Preparing Quality Assurance Program Plans" (QAMS-004/80) as published by the EPA's Quality Assurance Management Staff, Office of Monitoring Systems and Quality Assurance, Office of Research and Development.

Director of	Quality Assurance: Solut - Meren
	Pill Adams Domes & Macre
Date:	Tune 29 1988
Conv Number:	257

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AR300516

## QUALITY ASSURANCE PLAN

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# 1.0 QUALITY ASSURANCE PROGRAM PLAN IDENTIFICATION FORM

Document Title Quality Assurance Plan: CompuChem® Luboratories				
Document Control Number:				
Organization Title: CompuChem® Laboratories, Inc.				
Address: P. O. Box 12652				
3308 Chapel Hill/Nelson Highway				
Research Triangle Park, NC 27709				
Responsible Official: Mr. Ross Robeson Telephone: (919) 549-8263				
Title: Vice-President and General Manager of Laboratory Operations				
Quality Assurance Officer: Mr. Robert E. Meierer Telephone: (919) 549-8263				
Address: CompuChem® Laboratories, Inc.				
3308 Chapel Hill/Nelson Highway				
- Research Triangle Park, NC 27709				
Plan Coverage Environmental Laboratories Including: Production Planning and Control Glassware Preparation Sample Preparation Laboratory High-Hazard Laboratory Inorganics Laboratory GC/MS Laboratory GC Laboratory GC Laboratory Data Entry and Report Preparation Dioxin Coordination and Reporting EPA Technical Review EPA Customer Inquiry Commercial Technical Review Commercial Customer Inquiry Quality Assurance				

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Conc	urrences		Letter and Line Control of the Contr
(1)	Name:	Mr. Robert E. Meierer	
-	Title:	Director of Quality Assurance	
	ature:	Sold F. Merin	Date: February 2, 1987
{2}	Name:1	Mr. Ross K. Robeson	•
	Title:	Vice President and General Manager - I	Laboratory Operations
Sign	<pre> <pre> ature: _ </pre></pre>	XX Lolisson	Date: February 2, 1937

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#### 2.0 INTRODUCTION

CompuChem® is dedicated to providing the highest quality data available. In addition to a Quality Assurance Director, who is responsible for the overall quality assurance program at CompuChem®, the QA Department consists of Quality Assurance Specialists and support staff. The QA program meets or exceeds EPA recommended guidelines, with quality control samples accounting for at least 20% of the total number of samples analyzed. The Computerized Laboratory Management System (CLMS) automatically schedules the introduction of QC samples (spikes and duplicates), and internal performance statistics are determined quarterly on each test parameter, using the total sample data base. These data can be used to update control limits, or in the case of programs with defined control limits, the data serves to demonstrate overall lab performance.

Data are reviewed at three levels, including a final review by the senior technical staff, and a percentage data audit by the QA Department.

The lab must demonstrate that the analytical procedures and techniques are in control. This is established by the use of specified laboratory proficiency or method validation studies. These studies are fined in Appendix A. Once the studies are complete and the data have been assessed, normal QC activities are performed. These activities include duplicates, matrix spikes, blank spikes and the use of surrogates for all organic analyses, which evaluates total system control on a per sample basis.

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#### 3.0 QUALITY ASSURANCE POLICY STATEMENT

- Statement of Authority and Responsibility

This document is the Quality Assurance Plan for the Environmental Operations Division of CompuChem® Laboratories. The Plan describes the activities of the Division necessary to meet or exceed the data quality objectives of CompuChem's clients.

The Management of CompuChem® Laboratories is fully and firmly committed to the quality assurance program described in this Plan. Each director, manager, and supervisor as well as their staff members, as assigned in accordance with this Plan, are obligated to comply with its stated requirements, responsibilities, and objectives.

The QA program will be maintained and expanded or modified as necessary, to ensure all reportable data are of uncompromising quality.

The Director of Quality Assurance is responsible for the contents of the Plan and is committed to assuring that the stated requirements are met. The Director of Quality Assurance has the additional responsibility and authority to terminate nonconforming work.

Robert Meierer

Director of Quality Assurance

Ross Robeson

Vice-President, and General Manager of Laboratory Operations

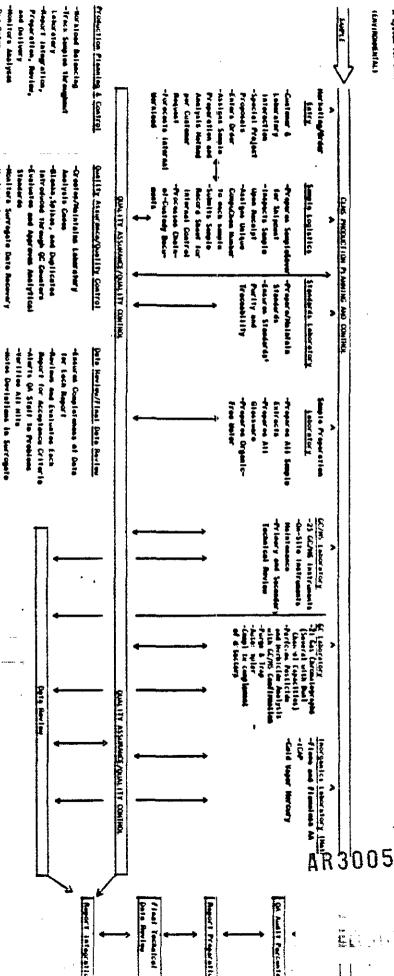
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#### 4.0 QUALITY ASSURANCE MANAGEMENT

# 4.1 <u>Introduction</u>

The Computerized Laboratory Management System (CLMS) Chart shown on the next page illustrates the interaction of quality control functions with all laboratory units. As shown, the Quality Assurance Department's staff monitors and reviews all laboratory units and operates independently of production areas.

A bysem for Embassed Quality Cameral and Sample Tracking in Volume Oriented Organic and Inurganic Analyses



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Report integration Report Preparatio Date Series final Technical

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#### 4.2 Assignment of Responsibilities

The Quality Assurance Department operates independently of all data generating areas. The QA Director reports directly to the President.

#### Roles and Responsibilities:

The main objectives of the laboratories' QA program are to assure that our laboratories generate high quality results, identify and implement policies to improve quality, and maintain the necessary records that document laboratory performance. The success or failure of the program depends on the people carrying out the various steps of the program.

A Listing of QA personnel responsibilities and authorities follows.

Responsibility and Authority of the QA Director:

To be certain that the laboratories achieve QA objectives, the Director of Quality Assurance monitors and directs the QA programs goals, in strict adherence to the procedures and requirements stated in this Plan.

The QA Director is independent of and separate from all personnel directly involved in the direction and operation of the technical program.

Additionally, the QA Director's duties include:

Monitoring the QA program as documented in the QA Plan and ensuring that the program is carried out.

Developing and implementing new QA programs, including statistical procedures and techniques.

Conducting regular audits and inspections, reporting the results to management, and when needed, ensuring that corrective action is taken.

Maintaining current copies of all measurement procedures routinely used in the laboratories, including subcontract laboratories.

Informing management of the status of the QA program.

Seeking out and evaluating new ideas and current pdevelopments in the field of QA and recommending means for their application wherever advisable.

In conjunction with his interactions with the Marketino Department, the QA Director advises Marketing on approcedures concerning sample analyses.

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The QA Director implements or modifies analysis codes and procedures as needed.

The QA Director has the final authority to stop or change any incorrect or improper sampling or analytical procedure to assure data/product quality.

## Responsibilities and Authorities of the QA Staff:

Spot-checking work in progress for quality and completeness.

Providing deviation reports to laboratory managers and the QA Director on out-of-control analyses and providing recommendations for corrective action.

Overseeing corrective action as required.

Assuming the responsibilities of QA Director, if necessary.

Ensuring that the laboratories meet all requirements as documented in this plan, as well as their specific SOP manuals.

Ensuring generation, analysis, and documentation of QC Data.

Establishing the control limits using QC data from routine analyses.

Providing information and documentation for audits or inspections.

Functioning as a liaison between the QA Director and personnel within the laboratories.

Communicating to the QA Director any quality problem or potential quality problem within the laboratories.

Writing QA notices for inclusion in data packages.

Conducting unannounced audits.

Reviewing and approving Performance Evaluation sample data before release to the client.

Coordinating projects with other QA staff.

Introducing internal "blind" check-samples into the system 526 and reporting their performance to management, including bline 6 performance checks of subcontractors.

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Ensuring that all standards are approved and traceable to standards provided by the National Bureau of Standards (NBS) or EPA.

## Responsibilities of Laboratory Personnel and Management:

Preventative maintenance, including routine and scheduled.

Compliance with methods as written.

Ensuring that the instruments meet calibration and tuning requirements.

Ensuring that instrument and calibration logs are maintained.

Responding to corrective action requirements.

Performance of action steps based on QC acceptance critera.

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#### 4.3 <u>Communications</u>

The Quality Assurance Department communicates to other areas of the laboratories and to Management via several different kinds of reports. The Director of Quality Assurance and the QA staff distribute memos to appropriate laboratory management detailing the results of internal and external audits, blind QC samples, and data audit reviews. These reports indicate that corrective action is needed, or in many cases they are used to reaffirm that the laboratory areas are performing in a satisfactory manner.

Every month the QA Department releases a report summarizing its activities during the previous month. Typically, the information in this report includes the results of internal and external audits, condition code reports, the labs' performance on internal blind QC samples, the labs' performance on external performance evaluation (PE) samples, summaries of special studies conducted, and summaries of any other activities conducted by the QA Department. This monthly report is sent to upper-level management and laboratory managers.

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V.P., FDT Warketing/Sales Mike Terretti President, Chemitest Analytical Labs Joel Bird Quality Assurance Robert E. Melerier Director, His Dan Kessier Director, Administrative Assistant Director, Environmental Debble Petillo Marketing/Sales Rick Gigilo Director, Environmentai Chief Executive Officer Testing Operations Koos Verkerk Koos Verkerk (Acting) V.P., Environmental Market Development Thomas A. Peacock Human Resources George Hedrick Olrector, General Manager Forensic Orug Testing Operations James McCarthy Paul Brunswick V.P. 4 CF0 AR300529

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Revision No. 4
Date: October 17, 1988

Manager, Increanics Labs | Manager, Organic Labs Steve Walburn Bruce Robribach Prop/Final Technical CompuCheme Laboratories, Inc. Review Ann Flaherty Manager, Environmental Testing October 17, 1988 Operations (Acting) Kees Verkerk Director Industrial Engineer (Vacent) Instrumentation Jim Chambers Meneger, Lab Director, Production, Pianning and Control Richard Bloom

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Date: October 17, 1988

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## 4.4 <u>Document Control</u>

Assurance Handbook for Air Pollution Measurement Systems, Volume I

(EPA-600/9-76-005), are used in the production of the QA Plan and other documents vital to the operation of the laboratories. This document control system includes distribution lists, a historical file of all updated standard operating procedures, and appropriate sign-offs for the ensurance of correct methods and techniques.

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## 4.5 QA Program Assessment

The Director of QA and the staff of that department conduct periodic assessments of the total QA program. Based on these assessments, a semi-annual written status report of QA activities and progress is forwarded to the President. These QA reports include such information as:

- 1. Status of or Changes to QA Program Plans
- 2. Status of QA Project Plans, if any
- 3. Measures of Data Quality
- 4. Significant QA Problems, Accomplishments, and Recommendations
- 5. Results of Performance Audits
- 6. Results of Systems Audits
- 7. Status of QA Requirements for Contracts and Grants
- 8. Summary of QA Training (internal and external QA/QC seminars)

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#### 5.0 PERSONNEL RESPONSIBILITIES AND QUALIFICATIONS

#### 5.1 Introduction

with over 240 employees, CompuChem® offers the scientific and technical expertise needed to service the analytical and informational needs of our customers. In addition to our skilled analytical laboratory personnel (with expertise in organic and inorganic analyses) CompuChem® utilizes a computer system staff that plans, develops, and implements software systems for data management and sample scheduling and control. To insure that the analytical needs of our clients are met, customer service representatives are assigned to each account, providing a liaison between the customer and the laboratory.

The following tables present the personnel of the Environmental laboratories by groups. Also, in Appendix B the resumes of all key personnel are presented by laboratory groups, and personnel requirements for EPA Contracts are listed.

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#### 5.2 Training

There are two types of training at CompuChem<sup>5</sup>. For new employees training consists of one-on-one training by the department manager, supervisor or one of our experienced technologists. This training follows an organized format with stated objectives and evaluations at various intervals.

The second form of training is for new procedures or new instrumentation as they arrive in the laboratory. The manufacturer usually provides training courses and certificates for those who successfully complete the program. These certificates are maintained in the employee's records. Supervisors and senior technologists who are trained by the manufacturer are then responsible for instructing and training other employees (and records are maintained on their training).

In addition to the initial training, employees are encouraged to participate in continuing education. The continuing education may be of several forms. Intradepartment short educational or review sessions are conducted by the managers or director of the department. A variety of local seminars, workshops, and lectures are made available to the employees. At the estimates of the employees on the content of these seminars in an in-house seminar. Other in-house seminars involve topics such as troubleshooting or recent developments that have appeared in the scientific literature.

Computher provides employees with an Educational Assistance Program. This program provides reimbursement for courses that enhance the employee's job performance and opportunities for advancement. AR300535

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## COMPUCHEM LABORATORIES, INC.

## 5.3 Environmental Operations Department Personnel

C. W. Bannerman

Vice President Environmental Operations

P. H. Mashburn

R. L. Bloom

Administrative Secretary Director of Production

#### Report Preparation/Report Deliverables

A. E. Evans

M. E. Mitchell

Y. L. Dunn

T. G. Hooper

C. P. Johnson C. B. McGhee

M. K. Murphy

J. D. Perkins

D. R. Byrd

D. K. Ramsey

A. M. Daniel

T. L. McQueen

S. D. Pierce

A. B. Spruell'

D. L. Jeanette

J. C. Garrett

C. M. Horton (2nd)

C. A. Keith (2nd)

M. D. Parks (2nd) M. Gibson (2nd)

Report Preparation Manager

Report Deliverables Supervisor

Deliverables Clerk

Data Entry/Report Integration Clerk

Quality Control/Report Integration Clerk Quality Control/Report Integration Clerk

Senior Data Entry Clerk

Deliverables Word Processor

Deliverables Word Processor

Technical Review Coordinator

Report Preparation Supervisor

Report Integration Clerk

· Senior Report Integration Clerk

Senior Report Integration Clerk

Quality Control/Report Integration Clerk

Acting Report Prep. Sup. (2nd Shift) Senior Data Entry Clerk

Deliverables Word Processor

Senior Report Integration Clerk

Deliverables Clerk

# <u>Production Planning and Control</u> /Scheduling and Sample Saver

A. M. Flaherty

P. J. Mock

C. S. Dover

L. B. Dickens (2nd)

M. A. Gabriel

L. F. Holloman

R. S. Oakley (2nd)

J. Morrisey (3rd)

Manager Production Planning & Control

Laboratory Production Coordinator

Laboratory Production Coordinator

Sample Custodian

Sample Custodian

Scheduling and Control Clerk

Scheduling and Control Clerk R300536

Sample Custodian

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K. A. Brady E. R. Nowell (2nd) J. P. McConney (2nd)

M. B. Odulana

P. H111

L. H. Jakes

C. T. Evans

T. R. Hux (2nd)

C. E. Howington

Supervisor Sample Saver & Scheduling

Scheduling Clerk Scheduling Clerk

Technical Reviewer

Technical Reviewer

Technical Reviewer

Technical Reviewer

Scheduling Sample Custodian

Technical Reviewr Traniee

#### LABORATORY OPERATIONS DEPARTMENT

#### C. A. Rezac

#### Director Laboratory Operations

## Sample Preparation Laboratory

M. L. Stanley

N. Her

M. K. Farmer

K. S. Hinshaw

A. L. Mitchell

A. D. Rice E. H. Thompson

C. Webb

C. Howell (2nd)
Y. Martin (2nd)
L. A. Pittman (2nd)

J. Venable (2nd)

Supervisor Sample Preparation Laboratory

Senior Laboratory Assistant

Senior Laboratory Assistant Senior Laboratory Assistant

Senior Laboratory Assistant

Laboratory Assistant

Glassware Preparer

Glassware Preparer

D. R. Stanley (2nd)
D. A. Caldwell (2nd) Supervisor Sample Preparation Laboratory
Senior Laboratory Assistant
Senior Laboratory Assistant
Senior Laboratory Assistant
Laboratory Technician

Glassware Preparer

#### GC/MS Laboratory

S. G. Walburn

B. H. Bell

E. S. Byrd

L. L. Fowler

G. M. Jordan

S. Maingi

C. T. Mann

D. B. Moore

G. Williams

J. Iqbal

Assistant Manger GC/MS Lab - Semivolatile GC/MS Operator

Senior Semi-Volatile Data Specialist Senior Semi-Volatile Data Specialist Senior GC/MS Operator

GC/MS Operator

Senior GC/MS Operator

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GC/MS Operator Trainee Laboratory Clerk

GC/MS Operator

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#### GC/MS Laboratory (cont.)

A. T. Chan (2nd) L. H. Bryant (2nd) P. B. Hopkins (2nd)

F. B. Littlejohn (2nd)

B. D. Livingston (2nd) S. Minor (2nd)

S. D. Wagner (2nd)

L. J. Wilkerson (2nd)

D. M. Alexander (3rd) S. R. Colemen (3rd)

V. D. Davis (3rd) M. Mattocks (3rd)

G. Mikhael (3rd)

M. A. Jackson (3rd)

M. Ramchandani (3rd)

Assistant Manager GC/MS Lab-Semi-Volatile GC/MS Operator Trainee GC/MS Operator Trainee GC/MS Operator Trainee Senior GC/MS Operator

GC/MS Operator

Senior GC/MS Operator

Laboratory Clerk

Supervisor GC/MS Lab-Semi-Volatile

GC/MS Operator

GC/MS Operator Trainee GC/MS Operator Trainee

GC/MS Operator

Laboratory Clerk

GC/MS Operator Trainee

#### Volatiles Laboratory

S. W. Bass

B. M. Barefoot (3rd)

C. D. Beck

K. E. Bonnell (2nd)

S. A. Hubbard (2nd)

G. R. Lambert

S. P. McCoy N. L. Moore (3rd)

T. C. Spruell

R. J. Pollock

L. R. Flynn

#### GC/DIOXIN PROGRAMS .

J. B. Henes

High-Hazard Laboratory

L. M. Sutton

B. H. Bell

M. F. Swift

. Manager Volatiles Laboratory

GC/MS Operator Trainee GC/MS Operator GC/MS Operator GC/MŠ Operator Trainee

Senior Volatile Data Specialist Senior GC/MS Operator

Laboratory Clerk

GC/MS Operator Trainee

Senior GC/MS Operator

Senior Systems Analyst Development Chemist

Director GC/Dioxin Programs

Manager Dioxin Programs Dioxin Data Coordinator -- Dioxin Data Coordinator

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## <u>High-Hazard Laboratory (cont.)</u>

J. Bumgarner (2nd)

D. K. Branoff (2nd)

M. L. Enscore (2nd)

V. Respass (2nd)

M. A. Riggs (2nd)

M. Ritchie

A. S. Thomasson (2nd)

Supervisor GC/Dioxin Sample Preparation Laboratory Chromatographer Trainee Laboratory Chromatographer Laboratory Chromatographer Senior Laboratory Assistant Laboratory Assistant Senior Laboratory Assistant

#### GC Projects

Ì

W. R. DesJardins

C. W. Abel (2nd) C. M. Dulaney

K. Hinshaw (2nd)

V. Barbour

N. R. Frank

D. P. McCormack

D. Studt (3rd)

# Inorganics Laboratory

B. J. Andershock (3rd)

J. W. Asprey

M. R. Grey (2nd)

S. Hashamu L. F. Jones D. C. Stogner J. C. Tzavaras (2nd)

B. Newton

Manäger GC Projects Senior Chemist GC Technician GC Technician GC Data Clerk Chemist Senior Chemist

Inorganics Technician Senior Chemist
Technician
Inorganics Technician
Junior Chemist

GC Technician

Senior Technician Senior Chemist Data Clerk

# QUALITY ASSURANCE

#### R. E. Meierer

W. J. Boone

W. Morton

D. G. Twine

R. J. Whitehead

R. V. Joshi (2nd)

# Director of Quality Assurance

Senior QA Specialist Communications Specialist Quality Assurance Clerk Senior QA Specialist QA Specialist

## LAB INSTRUMENTATION DEPARTMENT

J. T. Chambers

P. T. Williamson

J. Biggerstaff (2nd)

I. L. Gregory
D. L. Rich

T. Silver (3rd)

Manager Lab Instrumentation Staff Consultant AR300539 Electronics Technician

Senior Electronics Technician Senior Electronics Technici>-

Electronics Technician

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# **FACILITIES**

R. A. Parker
B. C. Allison
H. Brown
E. F. Floyd

Manager Facilities and Safety Facilities Maintenance Technician Maintenance Assistant Warehouse Facilities Assistant

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## 6.0 Facilities, Equipment and Services

## 6.1 Introduction

This section describes the facilities at CompuChem®, the instrumentation and peripheral equipment, and the services provided in maintaining the facility.

CompuChemo is located in Research Triangle Park, NC, 15 miles west of Raleigh. The total facility is comprised of both the Environmental and Forensic Drug Testing Operations of CompuChem® Laboratories, Inc. The two operations have separate laboratories that function independently, including separate computer systems. Much of the office space is also separate, however, many administrative functions overlap (i.e., Accounting, Quality Assurance, Human Resources, Computer Operations) and share common office space. Facility space allocation is described in section 6.2, and includes the Environmental Operations laboratory space, Environmental office space, and administrative office space common to both operations, totaling approximately 64,000 square feet. The two operations share two adjacent buildings which are connected by a permanent, enclosed walkway. Electrical power is supplied by Duke Power Company, with a service capacity of 2000 amperes at 480 volts. The enviornmental controls for the heating, ventilating, and air conditioning systems are Honeywell Electric and provide automatic starting and stopping as well as temperature control. All critical temperature areas such as refrigerators, freezers and computer rooms are monitored 24 hours/day by an off-site monitoring firm. The temperature of the refrigerators and freezers is maintained by a standby generator in the event of a power failure  $R_{ab} = 0.0541$ electrical power to the computer room is regulated by a power conditioner.

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Building security is maintained at all times. The main entrance is monitored by a full-time, contracted security staff (24 hours/day, seven days/week). Visitors must sign-in at the security guard's desk and be escorted through the facility by members of the staff. The exterior doors as well as the doors of various controlled access areas within the building are equipped with electronic card readers, controlled by Rusco Electronic Card Entry Access System. A burglar alarm system has been integrated with the Rusco system to provide protection when the facility is closed. Smoke detectors, as well as associated pull stations and fire alarm horns, are provided throughout the building for fire protection. Adequate fire extinguishers and emergency equipment are also provided. The fire burglar alarms are also monitored by the off-site security firm. When an alarm sounds, the off-site personnel alert the appropriate laboratory personnel, the Sheriff's office, or the Fire Department, as necessary.

CompuChem® Laboratories contains sophisticated, state-of-the-art instrumentation and data processing equipment capable of performing most organic and inorganic analyses. Two Hewlett Packard-3000 Series 70 mainframe computers are dedicated to scheduling and tracking sample analyses through the laboratories and are directly networked to GC/MS instrumentation. An HP-3000 Series 950 mainframe provides system redundancy in the event of primary system failure, and handles additional production coordination. One of two HP-3000 Series 39 microcomputers is dedicated to systems research; the second handles all accounting functions. The Computerized Laboratory Management System (CLMS) is accessed by laboratory, marketing, systems, and accounting personnel via more than 90 CRT AR 300542 terminals.

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The Manager of Facilities and Safety, Manager of Instrumentation, and Manager of Computer Operations are primarily responsible for the evaluation, selection and maintenance of all facilities, instrumentation, and computer equipment, respectively. The Manager of Facilities and Safety is also responsible for overseeing general housekeeping services and functions as the Laboratory Safety Officer. In this capacity, the Safety Officer conducts periodic safety inspections and manages the activities of the Safety Committee.

All analytical instruments are maintained by a staff of full-time service technicians, operating during all three shifts, seven days/week (also available on-call on weekends). Instrument log books are maintained for each individual instrument in each of the laboratories (GC/MS GC, Inorganics), for recording routine maintenance performed by the operator or laboratory staff.

Additionally, service records for each instrument are kept in the Maintenance Department to record all routine and non-routine maintenance performed by service technicians.

The Pure Water Room houses a state-of-the-art water purification system. Municipal water is fed through two mixed-bed ion exchange cylinders and a high capacity activated carbon tank. The effluent is pre-polished by two mixed-bed ion exchange columns, distilled in a Corning 12-liter all-glass still, then passes through a Megapure Polishing System. This final purification process feeds water through two more mixed-bed ion exchange cartridges, and activated carbon column and a clarifying filter. Water quality is monitored daily by an in-line specific conductivity meter, and by the various method blank and instrument blank QC checks performed on the water. A similar system is used at

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an off-site warehouse facility to produce pure water used in the trip blanks that accompany SampleSavers (sample coolers) into the field during sampling operations. The Sample Preparation Laboratory and QA SOPs include additional information regarding the operation of the stills.

Two other laboratories have systems in-place to perform additional processing of the water from the Pure Water Room. Teflon transfer lines feed the water into the Inorganics Sample Preparation Laboratory and Volatile GC/MS Laboratory systems. Inorganics Lab pure water passes through an additional Millipore Pure Water System (with ion-exchange cartridges and a carbon filter), and water for the Volatile Lab is sparged with nitrogen in an all-glass reservoir for 24 hours prior to use.

The laboratory also has a full complement of support equipment and instrumentation, such as glove boxes and hoods, walk-in refrigerators, freezer units, autoanalyzers, and sonicators.

The following sections describe the laboratory area by function and equipment. The floor plan was designed to allow for the efficient and secure movement of samples and data between work areas.

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#### 6.2 <u>Laboratory</u> Areas

Shipping and Receiving: This area is located adjacent to the laboratory section of the building. Samples arriving are identified and introduced into the scheduling and control system. The sample receiving area for environmental samples has about 2,450 square feet of floor space. The receiving area has 102 square feet of bench space for receiving and opening samples, three data entry stations, one laboratory sink and ample storage shelving. A 2,500 cubic foot refrigerator  $(4^{\circ}C \pm 2^{\circ}C)$  is provided for storage of environmental samples.

<u>Walk-in Refrigeration System</u>: This area is accessed from the shipping and receiving area as well as from the central laboratory corridor. This unit has two independent refrigeration systems, is temperature controlled to  $4^{\circ}C \pm 2^{\circ}C$  and is equipped with an activated carbon air filtering system, which maintains an environment free of organic vapors. The temperature is recorded daily. Both entrances are secured by locks and the temperature-activated alarm system is tied into a r ivate security service. In the event of unauthorized access or temperature factuations, appropriate parties are notifed by the private security service.

Extractions and Preparations Laboratory: This area is equipped with hoods as well as extraction equipment sufficient to process many thousands of samples per month. The environmental sample preparation laboratory has 2,024 square feet of space, two 8' fume hoods, three IEC centrifuges, two pactures 545 ovens, two sinks, six water baths, and 220 square feet of bench space. The air handling system for the sample preparation laboratory was custom designed.

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the extraction process. Conditioned 100% outdoor air is supplied into the room through linear diffusors and exhausted through exhaust ducts which extend from wall to wall on the north and south ends of the laboratory. This method maintains air flow at the workstations at all times and virtually makes the room a large walk-in fume hood. A complete air exchange occurs every two minutes. Separate exhausts are provided for furnaces and hoods. Adequate cabinet space is provided. Specially-designed water baths controlled and programmable to temperature and duration are also used. The glassware preparation room has 750 square feet of floor space and is equipped with two glassware washers, 26 feet of stainless steel counters with four built-in sinks, and one 72 cubic foot annealing oven.

<u>Solvent Storage Area</u>: This area is accessible only through a secured door adjacent to the extraction and preparation area. The room is designed with reinforced concrete walls, an automatic halon fire-extinguishing system, alarm systems and a roof that relieves pressure in the event of an accident.

GC Laboratory: The laboratory's twenty-one gas chromatographs are equipped with autosamplers or purge-and-tray devices (Tekmar LSC-2) and are interfaced with a Hewlett-Packard 1000 laboratory computer for data processing (all of which are installed on a raised computer floor). A variety of detectors are attached to the GCs, including Flame Ionization (FID), Flame Photometric, Electron Capture, Thermionic Specific (also called NPD or AFID), Photoionization (PID), and Electrocoulometric (also called a Hall Detector) detectors. AR300546

GC/MS Laboratory: The special features included in this area are numerous. All twenty-three GC/MS systems are raised on a computer floor. This allows gas, water, cooling and exhaust systems required to

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instrument to be introduced to the room independently, beneath the floor. Equipment is arranged in efficient working clusters. In this way, specific instruments can be utilized for specific types of analyses. For example, several instruments are totally dedicated to volatile organic analyses. These instruments are never subjected to semi-volatile work; therefore, cross-contamination of the instruments is eliminated. Furthermore, each cluster of instruments is staffed by experts familiar with the protocols associated with each specific procedure. This staffing system allows intimate daily interaction between the operator. his or her instruments and the methodologies required. All other instruments are dedicated in a similar fashion. Also located in a section of this area are two Hewlett Packard 3000 Computers used for support of scheduling and control activities and data networking. The combined GC/MS and Computer Room have a total of 3,380 square feet. Each GC/MS and computer is provided with an individual power supply from a breaker panel located within the lab. The GC/MS instruments are powered by three 1-phase, 75 KVA 480/220 volt isolation transformers. The computers are powered by one 3-phase 75KVA 480/208 volt isolation transformer. Helium, the carrier gas used, is supplied from a manifold system in an adjacent room through a piping system under the raised floor. There are three of these systems, each having a catalytic scrubber to remove traces of oxygen and water, prior to entering an instrument.

The 23 GC/MS instruments are configured with both packed and capillary GC columns, and have accessories for purge and trap, direct injection, or AR 300547 probe for introduction of samples. Both electron impact and chemical introduction sources are available. Each GC/MS instrument is equipped with its own dedirected microprocessor for data processing.

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Standards Laboratory: This area is separated completely from all other laboratories and is equipped with its own GC instrument. Refrigeration, glove box and hood units are located in this area. The entrance to this area is secured by two magnetic card locks and a cypher lock.

Inorganics Laboratory: This area is separated completely from all other laboratories and has one Inductively Coupled Plasma (ICP) unit, one Technicon autoanalyzer, two Atomic Absorption Spectrophotometers (AAS) instruments, and one UV/visible spectrophotometer. Several other analytical instruments required to perform classical analyses are also located in this laboratory. Hood systems are also an integral part of this laboratory.

In the Inorganics Sample Preparation area, there are 12 distillation units for cyanide and 6 units for phenol distillation. Mercury is detected by flameless-cold-vapor methods established by the USEPA (Cold Vapor Technique). For maximum data management, the Inorganics Laboratory uses a mini-computer (Digital, PDP11/73) interfaced to the ICP instrument (Jarrel Ash, Model 1100).

Extract Storage: Sample extracts are stored in spiritally-designed refrigeration units located adjacent to the Extraction Luboratory. These refrigeration units are accessed on a limited basis by a sample custodian only. Entrance is on a "need only" basis and requires a key to gain entrance. These refrigeration units are also connected to an alarm system. In the event of temperature fluctuations outside acceptable levels (4°C  $\pm$  2°C), appropriate parties are notified by a private security service and the problem is corrected by laboratory staff.

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High Hazard Laboratory: A limited access laboratory has been designed for sample preparation aspects associated with high-hazard samples. For example, all samples requiring analysis for 2,3,7,8-TCDD are prepared in this lab. Direct access to the laboratory is by means of a cypher lock. The hoods employe are equipped with a HEPA filtration unit. Laboratory personnel use more protective clothing than the other extraction laboratory personnel (i.e. full sack suits, booties, face masks, etc).

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### FACILITY SPACE ALLOCATION

# \_ \_ TOTAL LABORATORY BUILDING SQUARE FEET 24,005

1.	Sample Receiving	1,570 sq. ft.	
2.	Glassware Prep	750 sq. ft.	
3.	Organic Extractions and Inorganics Sample Preparation	2,008 sq. ft.	
4.	High Hazard Lab	450 sq. ft.	
5.	GC/MS	2,840 sq. ft.	
6.	Computer Room	1,450 sq. ft.	
7.	Standards Laboratory	312 sq. ft.	
8.	Metals (Indrganics) Instrumentation Lab	650 sq. ft.	
9.		1,200 sq. ft.	
10.	Solvent Storage	•	
11.	Utility	960 sq. ft.	
12.	Walk-In Refrigeration System_(2 units)	250 sq. ft.	
13.	Miscellaneous (Canteen, Corridors, Rest Rooms, etc.)	5,000 sq. ft.	
14.	Office*	6,023 sq. ft.	
	TOTAL PAMLICO BUILDING SQUARE FEET	55,487	
1.	Office*	_40,142 sq. ft.	

TOTAL COMPUCHEM LABORATORIES, INC. FACILITIES
RESEARCH TRIANGLE PARK, NC\* 79,492 sq. ft.

<sup>\*</sup> includes both Environmental and Forensic Drug Testing Operations.

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# GAS CHROMATOGRAPH LABORATORY EQUIPMENT

			······		- <del> </del>	-
Item	Model#	Serial#	CChem#	A-D#	Type ·	Installed
GC GC GC	VARIAN 3700 VARIAN 3700 VARIAN 3700 VARIAN 3700	58760308-13 71280469-13 32968966-11 74550509-13	000000	2&3 7&1 23	DUAL ECD AUTOSAMPLER DUAL ECD AUTOSAMPLER FID NPD FID	1980 1980 1980 1982
GC	HP 5880	2236A04163		21	FID	1982
60000000000000000000000000000000000000	VARIAN 3400 VARIAN 3400 VARIAN 3400 VARIAN 3400 VARIAN 3400 VARIAN 3400 VARIAN 3400 VARIAN 3400 VARIAN 3400	2006 2310 2309 2312 3623 3052 2308 2307 2311	001177 001175 001178 001173 001174 001179	5 0 4 6 9 10 12 14 24	FPD ECD NPD AUTOSAMPLER ECD NPD AUTOSAMPLER ECD FID AUTOSAMPLER ECD AUTOSAMPLER	1986 1986 1986 1986 1986
GC	VARIAN 3400 TEKMAR LSC-2 TEKMAR ALS 0.I. 442	3053 144 1016	001357 001647	19	HALL DET PURGE AND TRAP AUTOSAMPLER	1985
<b>G</b> C	VARIAN 3400 - 4460 HNU PI-52	3054 171-6-9B 620045	001356 001499 001362	<b>2</b> 0	PID DET PURGE AND TRAP	1985
GC	VARIAN 3400 TEKMAR LSC-2 TEKMAR ALS HNU PI-52	2306 1821 1041 620100	001176 001241 001648	18	PID PURGE AND TRAP AUTOSAMPLER	1985
GC	VARIAN 3400 TEKMAR LSC-2 TEKMAR ALS 0.I. 4420	2005 1556 902 6644-5-102	000953 001316 001649	17	HALL PURGE AND TRAP AUTOSAMPLER	1985
GC	VARIAN 3400 0.I. 4460 0.I. 0.I. HNU PI-52	3055 521-6051C 365-6-0020 05836	001358 001507 001508 001509	16	PID PURGE AND TRAP LOOP SAMPLING MODULE	1985
OVEN	BLUE M SW-11TA-1	SW365	001353		OVEN AR300	1551
COMP	UTER HP 1000				ALS SYSTEM DATA PROCESSING	, o o i

CHARCOAL AIR FILTERING SYSTEM

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# GC/MS LABORATORY EQUIPMENT (ENVIRONMENTAL)

OWA #	Serial#	Type Of Application	Installed
OWA - 1020	12137-0980	CAPILLARY COLUMN	9/81
OWA - 1020	12391-3-0281	CAPILLARY COLUMN	9/81
OWA - 1020	12141-0980	VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020	12138-0980	CAPILLARY COLUMN	9/81
OWA - 1020	12140-0980	CAPILLARY COLUMN	9/81
OWA - 1020	11957 <i>-</i> 2 <b>-</b> 0180	CAPILLARY COLUMN	9/81
OWA - 1020	_11957-3-0180	CAPILLARY COLUMN	9/81
OWA - 1020	11957-4-0180	CAPILLARY COLUMN	9/81
OWA - 1020		VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020	11957-1279	VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020 -	12391-2-0280	VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020	<b>1</b> 2391 0281	VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020	12139-0980	VOA-LSC/PURGE AND TRAP	<b>9/</b> 81
OWA - 1020	12391-1-0380	VOA-LSC/PURGE AND TRAP	6/82
OWA - 1020	12391-4-0381	CAPILLARY COLUMN	9/81
OWA - 1020	12391-5-0381	CAPILLARY COLUMN	6/83
OWA - 1020	12645-1-1181	VOA-LSC/PURGE AND TRAP	6/83
OWA - 1020 _	12645-4-1181	VOA-LSC/PURGE AND TRAP	6/83
OWA - 1020	12645-6-1281	CAPILLARY COLUMN	6/83
OWA - 1020 -	12645-3-1181	CAPILLARY COLUMN	6/83
OWA - 1020	12645-2-1181	CAPILLARY COLUMN	6/83
OWA - 1020.	\$12645 <b>-</b> 5-1281	VOA-LSC/PURGE AND TRAP	6/83
INCOS 50	13954-0387	HP-GC WITH CAPILLARY COLUM	N 1987

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### INORGANIC LABORATORY EQUIPMENT

<u>Item</u>	<u>Make</u>	Mode 1#	Serial#	Installed
AUTO ANALYZER II	TECHNICON	TRAACS 800		1987
CIRCULATING BATH	PRECISION			1987
ANALYTICAL BALANCE	METTLER	MODEL HL 52	A76373	<b>19</b> 80
ICP	JARRELL ASH	MODEL 1100	22483	<b>19</b> 86
MICROPROCESSOR IONALYZER PH METER	ORION	ORION 901	93353	1979
UV VISIBLE SPECTROPHOTOMETER	.VARIAN CARY	219	0438812	1981
CYANIDE/PHENOLS AUTOANALYZER	TECHNICON	AAII	<b>G</b> G0797940	1980
ATOMIC ABSORPTION SPECTROPHOTOMETER	INSTRUMENTATION LABORATORY	VIDEO 22(857)	<b>2</b> 027	1987
ATOMIC ABSORPTION SPECTROPHOTOMETER	INSTRUMENTATION LABORATORY	VIDEO 22(857)	2127	<b>198</b> 6
ATOMIC ABSORPTION SPECTROPHOTOMETER	INSTRUMENTATION LABORATORY	VIDEO 12(857)	2128	1986
VAPOR GENERATION ACCESSORY	AVA	440	1625	1986

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# 6.4 <u>Instrument Maintenance</u> .....

Analytical instruments are maintained by experts employed by CompuChem® on a full-time basis. Preventative maintenance as well as major instrument repairs can be accomplished on-site. An extensive in-house stock of spare parts allows for rapid repair. CompuChem® maintains service agreements with instrument manufacturers to further assure the operational viability of all in-house equipment.

The operational condition of instruments is one of the keys to successful completion of analytical tasks. This requirement is further magnified by the necessity to complete large programmatic requirements in a limited period of time. CompuChem's commitment to instrument maintenance assures clients that equipment will be available to generate the required data.

In discussing instrument maintenance services at CompuChem®, a distinction between GC/MS instruments and other hardware is required. In the case of the GC/MS instrumentation, CompuChem® staff have full maintenance and repair responsibility. These staff have been trained by the instrument manufacturer and are fully qualified to perform the required work. For other instruments, we have service contracts for periodic maintenance visits by the vendor, although maintenance personnel do assess whether repairs can be made in-house before outside vendors are called.

All GC/MS instrument repair logs and instrument service records are maintained in individual instrument files in the instrument repair shop.

AR300554

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All services performed on the instruments are recorded and filed on an instrument-specific basis to maintain an on-going historical record of the date and type of work performed. Similar records are maintained for preventative maintenance activities. Example 1, beginning on the next page of this section, shows a typical maintenance record for the GC/MS instruments.

A procedure manual outlining the proper use of each piece of equipment is maintained. These manuals are located with the instrumentation and include instructions for use, calibration, and maintenance of the instrument.

AR300555

				2					•
		CO#40	nonem ec	ZMS SERVI	COMPUCHEM CICINS SERVICE REPORT		<b>3</b> 2	्र इंड इंड	:
INSTRUMENT NO 1 2 DATE 0 7	2 3 8 6	ĭ K	1 1 5 1		OPERATOR: Susan Bass	Bass		005	;   1
GC oven not heating								R 3	
								Ā	
Annual control of the									-
START DATE 0 7 2 3 8 6	END DATE	0 7	2 3	8 6	RESPONSE TIME	5	S/E Lee Gregory	egory	
START TIME 1 0 1 5	END TIME		<u>-</u> 0	5   7	TOTAL DOWNTIME	1 2	JOB COMPLETE? YES	×	క
ASSEMBLY NO.	REV	CODE	FAILURG CODE	DESIGNATOR	OR PART NO.		DESCRIPTION	QTY	ωsτ .
7 0 1 7 8 9 1 4 3 2 0	<u>o</u>	0 4	0 6	 		11444	Fuse		1 35
	_								-
	_							_	
	<u> </u>								
COMMENTS: Oven not heating, 24 voit relay not engaging tound tuse F4 blown in power supply.	t relay no	† engagi	ng tound	tuse F4	blown in power	1	Replaced fuse F4		
checked +24 volts, checked operation of $6C_{ m p}$ gave back to operator L, $G_{ m p}$	ed operati	on of GC	gave b	ack to op	erator L.G.				
OPERATOR ACCEPTANCE;			0 31VG	0 7 2 3 8 6	8 6	341.1	HE 1 1 0 0 REV07298216	EV07298216	

· House

#### EXAMPLE 1 (CONTINUED)

#### INSTRUCTIONS

INSTRUMENT NO. - LIST AS 05 FOR DWA05, 12 for OWA12, ETC. THE 4021 GC/MS/DS IS INSTRUMENT 00. ALL STANDALONE DATA SYSTEMS ARE INSTRUMENT 99.

DATE & TIME -

ENTER DATE AS MM/DD/YY; AUGUST 28, 1985 IS 08/28/85. ENTER TIME BY 24-HOUR CLOCK. 9:25AM IS 0925 AND 9:25PM IS 2125. THE TIME AND DATE SHOULD BE WHEN A PROBLEM IS DISCOVERED AND REPORTED VIA THIS FORM.

\_ . . . . .

OPERATOR -

WHO YOU ARE.

PROBLEM CODE

4 DESCRIPTION - USE THE 3 DIGIT PROBLEM CODE THAT MOST APPROPRIATELY DESCRIBES YOUR PROBLEM. PLEASE DETAIL THE PROBLEM AS FULLY AS YOU CAN.

. USE BLACK INK ONLY & WRITE OR PRINT LEGIBLY.

PROBLEM CODES	}
P.M.	000
( NNOT MEET TUNE	001
IDOS ERRORS - LIST AND FULLY E SCRIBE WHAT TO DATA SYSTEM WAS DOING	002
NSITIVITY	003
TIMES	004
F SPECTRA OR N. MS RESPONSE	005
SOFTWARE NOMALIES	006
6S CHROM.	007
DISC DRIVE	800
FINTER	009
PURGE & TRAP	010
COUM FAULT	011
AIR LEAKS	012
" RMINAL	013
DATA SYSTEM	014
I NNOT BOOT	015
₽#KNOWN	016

REPAIR ACTION CODES		FAILURE ANALYSIS CODES
PIRATE PARTS	100	UNKNOWN 200
ADJUSTMENT - ELEC	102	MISCELLANEOUS 202
ADJUSTMENT - MECH.	104	OPERATOR ERROR 204
REPLACED ASSY.	106	SOFTWARE 206
		HEADCRASH 208
RETURNED TO VENDOR REPAIR	10B	MECH. DEFECT 210
RETURNED TO VENDOR WARRANTY	110	OUT OF ADJUSTMENT 29*
REQUESTED IN-HOUSE VENDOR SERVICE	112	INTERMITTANT 29*
WAITING FOR PARTS	'	EXCESSIVE NOISE 29*
(NOTE P.O. #)	114	EXCESSIVE WEAR 212
CLEANED SEPARATOR ' .	116	SHORTED COMPONENT 214
CLEANED MASS FILTER	118	OPEN COMPONENT 216
CLEANED SOURCE	120	FAULTY CRIMP 218
REPLACE PART	122	POOR CONTACT 220
REPAIR IN-HOUSE	124	POOR SOLDER JOINT 222
UNABLE TO REPRODUCE	126	DIRTY/DUSTY 224
		LEAKING 226
		REPLACE . WITH
		1 = ELECTRICAL 2 = MECHANICAL 3 = VACUUM 4 = SOFTWARE

AR300557

### PREVENTIVE MAINTENANCE - 3 MONTH INTERVAL

### REPAIR - PREVENTIVE MAINTENANCE CHECKS AND SERVICES GUIDE"

	SERVICE INTERVAL								
TEMS TO BE INSPECTED	PROBABLE SYMPTOM							SERVICE Internal	PROCEDURE
		dally	bl-monthly	3 months	6 months	بمعداد	es required	NOTE: Applicable are preented in Operator Menual(cotherwise specif	the Finnign s), unless
Sīgma 3 90									
1. Line fuses for the GC	inactive GC, blown fuse	$\vdash$					x	replace fuse	
2. Injector for packed columns							X		
3. Splitless injection for capillary columns							×		
4. Injector septum in the GC	obstruction, leaks	×					×	clean, Inspect or	
5. Ceriler gas com- nections/couplings 5. Ceriler gas filter	leakage Feplace when new gas	$ldsymbol{ldsymbol{ldsymbol{eta}}}$	<u> </u>			_	×	replace as required	
In the SC 7. Filter, flow	cylinder is installed dirty filter	<u> </u>				_	×	replace fliter	
controller 8. Cepillary column		<b>-</b>	<u> </u>	×		<u> </u>		clean Inspect of	
9. Packed column	excessive usage, leaks	├-	-			_	×	replace as needed	
(glass type) 10. Packed column	at injection and inter- face port of the zone-	-	-			-	X		
(metal type) 11. Detector port to	heating block	<del> </del>	-			$\vdash$	×		
907MS interface 12. GC cool down fan		i,		×		-	Ĥ	Inspect and/ or replace	

AR300558

These maintenance procedures meet or exceed finnigan's recommended preventive maintenance checks and services.

	1			SERV INTE	ICE RVAL	·			
ITEMS TO BE INSPECTED	PROBABLE SYMPTOM	,	(-month)y	months	acart he	<u> </u>	required	SERVICE INTERNAL	PROCEDURE
Ness Spectrometer		delly	b l-mor	2 70			88 F60		
1. Glass jet separator 2. Glass jet separator ferrules	obstruction or glass breakage						X	clean or replace	
3. Mass analyzer head assembly (in the vacuum manifold)	gross leaks, presistent pressure due to degesing of trapped gases in the vacuum system						×	Inspect	
*Megnet well flange assy *CAL gas valve assy *vent valve assy *water flow sensor switch	leakage, faulty CAL gas pressure (see Pirani gauge) faulty switch						X X X	Inspect Inspect Inspect	
4. Quadrupole mass enalyzer 5. Electron multiplier 6. Alcate! vacuum pumps (2)	•			X			×	Inspect and/ or replace	
7. Prolifer turbo pump Balzer turbo pump	dirty of I			x				end replace	Beizer Manual
Vecuum system filter/drier 10- ion Source	excessive use, dirty filter			x			χ.	clesn & Inspect	pg23
• ion source filement essy • collector • iens • aperture • ion volume	iack of sensitivity, faulty peak shape, no autofune	wit	h ev	er y	eurr 111a	ent I	•	clean, inspect or replace as required	
80/MS interface Oven 1. Capillary interface tubing 2. Separator divert fitting 3. Vacuum divert valve	p i ugged			×			x x x	clean, inspect and/or re- place	
Power Module  1. MS power supply  2. Turbo power supply				×			, X	measure & verify PCB	
Card Cage Module  1. Air filter at pottom of cage  2. Fan	dirty filter, obstruction of air flow burned out fan			×			×		
Bignat cable on Digital 170 PCB			,	<del> </del>			×	AR3005	59 

### REPAIR - PREVENTIVE MAINTENANCE CHECKS AND SERVICES QUIDE (Conf.)

		$\prod$		SERV INTE				·	
ITEMS TO BE INSPECTED	PROBABLE SYMPTOM		th I y	£	£	<u>.</u>	required	SERVICE Internal	PROCEDURE
		delly	bi-monthiy	3 months	6 months	yearly	es req		
Nova Computer								inspect end/or replace	
1. Fen	faulty fan rotation	+-		٠,		-		reprece	
Perkin-Einer Disk Drive		-		X	-	<b>-</b>			
1. Output signal					-			check and	
2. Adjustable DC voltages (+5Y, +13Y, -13Y)				×				verify	
3. Brushes								clean and/ or replace	
4. Positioner carriage guide					X			clean and inspect clean and	
Spindle chuck and cone		-			×	<u> </u>		Inspect Inspect	P/ECEM Manual
destite heads				×		<u> </u>		and repair	P/ECEM Manual
7. Fixed disk				×		<u>L</u> _			
8. Air filter		L				<u>  ×</u>			P/ECEM Manual
*prefilter *main filter				××		<u> </u>		replace replace	
9. Blower ground brush						×		replace	
.C. Spindle ground brush						x		replace	]
11. Blower drive belt								replace	P/ECEM Manual

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#### 7.0 DATA GENERATION

### 7.1 Quality Assurance Project Plans

When QA Project Plans are required for a specific project, they contain the following, as applicable:

- o Title Page, with provision for approval signatures
- o Table of Contents
- o Project Description
- o Project Organization and Responsibilities
- QA Objectives for Measurement Data, in terms of precision, accuracy, completeness, comparability, and representativeness
- Sampling Procedures
- Sample Custody
- o Calibration Procedures and References
- Analytical Procedures
- o Data Analysis, Validation, and Reporting
- o Internal QC Checks
- Performance and Systems Audits
- Preventive Maintenance Procedures and Schedules
- Specific Procedures to be used to routinely assess data precision, completeness, accuracy, comparability, and representativeness of specific measurement parameters involved.
- o Corrective Action
- QA Reports to Management

QA Project Plans provide for the review of all activities which could directly or indirectly influence data quality and the determination of those 53. operations which must be covered by SOPs. Activities to be reviewed may include:

- o General Network Design
- o Specific Sampling Site Selection

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- o Sampling and Analytical Methodology
- Probes, Collection Devices, Storage Containers, and Sample Additives or Preservatives
- Special Precautions, such as heat, light, reactivity, combustability, and holding times.
- Federal Reference, Equivalent or Alternate Test Procedures
- Instrument Selection and Use
- Calibration and Standardization
- Preventive and Remedial Maintenance
- Replicate Sampling
- o Blind and Spiked Samples
- o Collated Samplers
- QC Procedures, such as intralaboratory and intrafield activities and interlaboratory and interfield activities.
- o Documentation
- Sample Custody
- o Transportation
- o Safety
- o Da. Handling Procedures
- D Service Contracts
- o Measurement of Precision, Accuracy, Completeness, Representativeness, and Comparability
- Document Control

QA project plans are prepared in document control format, with provision for revision, as needed, and with a record of the official distribution 300562

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The quality requirements of proposal requests from prospective customers shall be identified upon the initial review and evaluation of the requests. When the quality requirements have been identified, the designated QA staff member shall ensure that they are adequately addressed in the Project Plan.

The following are Quality Assurance Program Objectives to be met as a project becomes operational:

- 1. Development of a QA Project Plan for the project, if required by the customer, or advisable per management request.
- 2. Assignment of responsibilities for achieving the required quality of materials, services, and quality assurance.
- 3. Organizing and staffing appropriately to implement quality assurance activities.
- 4. Development of working plans and procedures to implement the Quality Assurance Project Plan.
- Implementation of the QA Plan.
- 6. Coordination of QA activities with the customer, subcontractors, suppliers, etc.

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### 7.2 Standard Operating Procedures

SOPs are developed and used to implement routine QC requirements for all monitoring programs, repetitive tests and measurements, and for inspection and maintenance of facilities, equipment, and services.

The Environmental Laboratories' procedures are documented by two separate SOP manuals; the Sample Preparation Procedures (SPPs) and Instrument Procedures (IPs) are contained in one volume. The non-analytical Standarding Operating Procedures (SOPs) are contained in a two volume set, the SOP Manual: Environmental.

The indices for both procedure manuals (SPPs/IPs and non-analytical SOPs) are included in Appendix C. These indices should provide a good understanding of how thorough the documentation of procedures is at CompuChem; the documentation of procedures is considered to be very important to the ensurance of data quality.

Standard Operating Procedures (SOPs) are distributed by area; each section of the Environmental Laboratories receives its own set of SOPs. Complete copies of the SOP Manual are maintained in the library and the Quality Assurance office.

The organization of the SOP Manual reflects the progress of a sample through the laboratories. For instance, a sample first arrives in the receiving area (SOPs included in Production Planning and Control); it is prepared as necessary for analysis (SOPs included in the Sample Preparation Laboratory); it is analyzed as necessary (included in separate sets of laboratory 50Ps); the data is then prepared, reviewed, and reported, as indicated.

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If a question arises concerning the procedure followed for an activity in one of these areas, the SOPs for that particular area are consulted to resolve the question. These SOPs are also a valuable source of material for training purposes.

Completing the Initial Documentation Form: Each set of SOPs is accompanied by an Initial Documentation Form. This form is located at the end of each separate set of SOPs and serves as the procedures' sign-off documentation, indicating that the procedures are consistent with current laboratory practices. After the specific laboratory manager has determined that the procedure/s is accurate, he/she signs the Initial Documentation Form for those procedures and returns a copy to Quality Assurance.

The Initial Documentation Form is also used to ensure that personnel understand the tasks and responsibilities of their positions. All personnel review the SOPs for their positions and, provided they understand what they are responsible for, sign a copy of the Initial Documentation Form. The appropriate manager documents this understanding, after infirming that the employee does understand, by co-signing the form. Copies the completed form are forwarded to Quality Assurance and are filed in the employee's folder in the Human Resources Department.

Revising and Creating Standard Operating Procedures: Standard Operating

Procedures are updated as laboratory procedures change, and it is often
necessary to create new SOPs, as new procedures are developed to meet the information needs of CompuChem's clients. The current procedures for revising and
creating SOPs (Quality Assurance SOPs 3.2 and 3.3) can be found in the back of
each section of SOPs.

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Quality Assurance supports all sections in the developing, writing, editing, revising, and maintaining of current, accurate operating procedures.

All procedures remain the property of CompuChem® Laboratories.

All procedures that go outside the laboratories are CONFIDENTIAL.

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### 7.3 Additional Laboratory Polices to Achieve QA Objectives

Sample Preparation: The quality of work in the sample preparation area is crucial to the overall quality of the CompuChem® service. Before beginning the preparation of samples, a technician must demonstrate his or her proficiency with the procedure. This can be done by analyzing or preparing samples to produce results which can be compared and evaluated against established criteria. The management of the Sample Preparation Laboratory maintains records of such proficiency tests, and those qualified to perform certain procedures are specified in the Initial Documentation Forms of the area's SOP manual, which also become a part of the indivduals personnel file. Blind samples are submitted for continuing evaluations of the analyst's performance. The goals that can be measured are to produce or demonstrate acceptable recoveries of spiked compounds from samples, show no sample contamination during processing, provide proper documentation with an analysis, demonstrate precise and reproducible work, and show the exercise of correct technical judgement and abilities.

A minimum of 3 surrogate standards are added to each organic plane requiring GC/MS analysis for volatiles, acids, and base neutrals.

For pesticide and herbicide analysis, one surrogate is added for each. These surrogate standards are quantitatively analyzed in the GC/MS or GC phase.

Historical records are maintained on the percent recovery of surrogate standards for each sample and each analyst. These data form the statistical basis upon which preparation technique is monitored. Surrogate recoveries must AR300567 tance criteria before the analytical data will be released. In some increases.

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the sample matrix may produce interferences which adversely affect recoveries. These interferences must be confirmed by a repreparation and reanalysis of the sample; affected data are qualified by a Quality Assurance Notice.

With each new lot of reagents, a reagent blank is prepared and analyzed to assure that reagents do not introduce contaminants or interferences. A method blank is prepared at a frequency of at least one for every twenty samples processed for each analysis requested. The purpose of the method blank is to ensure that contaminants are not introduced by the glassware, reagents, personnel, or sample preparation environment.

Standards: Calibration standards are traceable to the National Bureau of Standards (NBS) or EPA whenever such standards are available. Commercial sources of standards and reagents are checked for purity, and approved, prior to their use in analysis.

All standards prepared for use throughout the laboratory are assigned a code number. The standard code number is entered in a bound standard notebook with all information regarding the preparation of that standard, i.e., date, technician, name of each compound and amount used, final volume, and solvent used. All standard containers are labelled with the standard's identification, lot number, code, manufacturer, and date.

The instrument response obtained for each compound in a newly prepared standard is compared to the response obtained from the previous standard. The two standards must agree within 15% (for all but a few compounds recognized as AR300558)

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being chromatographably atypical) or the new standard may not be used until the discrepancy has been resolved. The working lifetime of standard preparations are dependent upon the compound types comprising the standards. Shelf-life of standards is determined during storage stability studies carried out by the Standards Laboratory.

<u>GC/MS</u>: The Gas Chromatograph/Mass Spectrometer analysis is extremely important to the overall accuracy and precision of the CompuChem® service. To assure that the results from this phase are of the highest quality, a rigorous program of calibration and quality assurance has been established.

Instruments are calibrated before being put into service. Instruments must be recalibrated at regular intervals specified or approved by the accrediting body, and consistent with the manufacturer's recommendations. Instrument response is subjected to checks between the regular recalibrations. The nature and frequency of such checks are specified in the Instrument Procedures. The laboratory maintains adequate records of all calibrations, recalibrations and in-service checks of instruments. The schedule of checks depends on the experience of the laboratory's maintenance needs. All calibrations are traceable to primary standards of measurement. Where the concept of traceability of measurements to primary standards is not applicable, the laboratory provides satisfactory evidence of correlation or accuracy of test results.

Analysts, assistant managers, lab managers, and QA staff inspect all calibration data for completeness and validity. Forms are checked for arithmetic and procedural errors. Recurring errors, caused either by  $\frac{1}{4}$ R $\frac{300569}{00569}$  operators or by ambiguously worded instructions, are brought to tion of the department senior laboratory staff or laboratory materials.

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The mass spectrometer must first be calibrated according to the manufacturer's procedures using FC-43.

Once per shift the instrument is fine tuned using Decafluorotriphenylphosphine (DFTPP) or Bromofluorobenzene (BFB), depending on the use of the
instrument. The mass spectrum from DFTPP obtained should meet the criteria
described by the USEPA Caucus Organics Protocol of the Contract Laboratory
Program (CLP), or that specified in the Federal Register (October 20, 1984).
For DFTPP, the key ion and ion abundance criteria are:

TABLE I

Ion Abundance Criteria
30-60% of mass 198
less than 2% of mass 69
less than 2% of mass 69
40-60% of mass 198
less than 1% of mass 168
base peak, 100% relative abundance
5-9% of mass 198
10-30% of mass 198
1% of mass 198
less than mass 443
greater than 40% of mass 198 AR 300570
17-23% of mass 442

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When volatile organics are analyzed, DFTPP cannot be used because of its low volatility. In these cases, Bromofluorobenzene (BFB) is used. The key ion abundance criteria are:

TABLE II

m/e	Ion Abundance Criteria
50	15-40% of the base peak
<b>7</b> 5	30-60% of the base peak
<b>9</b> 5	Base Peak, 100% relative abundance
96	5-9% of the base peak
173	Less than 1% of the base peak
174	Greater than 50% of the base peak
175	5-9% of mass 174
176	Greater than 50% of the base peak
177	5-9% of mass 176

Once the instrument has met key ion abundance criteria for the above mentioned compounds, it is calibrated. Calibration curves are generated as outlined in the Caucus Organics Protocol, (Rev. 1985), and in the Federal Register (October 26, 1984).

<u>Calibration of the GC/MS System</u>: After the master set of instrument calibration curves has been established, they are verified each shift by injecting at least one standard solution. If significant drift has occurred, a new calibration curve must be constructed. The drift is defined in either EPA's requirements as specified in the CLP or in the Federal Register (October 26, 1984).

Inorganics: Metals, except mercury, are analyzed using flame and furnace AAS and ICP spectroscopy. The analysis procedure involves two steps: digestion and subsequent instrumental analysis. The quality of these results is assured, by several key procedures.

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For each batch of samples in the digestion process, a method blank is included. This blank is analyzed along with the samples to assure there were no contaminants introduced by the reagents or laboratory procedures.

Calibration of the AAS and ICP Systems

For inorganic analysis by AAS and ICP, initial calibration is performed using dilutions of stock metal solutions. For AAS calibration, a blank and at least three calibration standards are employed. For ICP analysis a mid-concentration level standard is analyzed. Prior to the ICP calibration and on a quarterly basis, a linear range verification check standard is analyzed for each element. The analytically determined concentration of the standard must be within 5% of the true value. This concentration, then, is the upper limit of the ICP linear range. Results cannot be reported beyond that upper concentration level unless they are a result of an appropriate dilution/reanlysis.

After the AAS and ICP systems have been calibrated for every analyte, the initial calibration is required to be verified for accuracy. This is accombished by immediately analyzing on EPA Initial Calibration Verification Solution or any other independent standard at at concentration other than that used for calibration, but with the calibration range. An independent standard is one composed of the elements from a different source than those used in the initial calibration.

In order to assure calibration accuracy during the course of sample analyses another QC sample, a Continuing Calibration Verification Standard, is analyzed at a frequency of 10% or every 2 hours during the analysis runk Roscato 72

analyte. The analyte concentrations in Continuing Calibration Verfication

Standard are near the mid-range level of the calibration curv. The Initial and

Continuing Calibration Verfication Control Limits are:

INITIAL AND CONTINUING CALIBRATION VERIFICATION CONTROL LIMITS FOR INORGANIC ANALYSES

Analytical Method	Inorganic Species	% of True Vi Low Limit	alue (EPA Set) High Limit
ICP Spectroscopy/ Flame Atomic Absorption Spectrometry	Metals	90	110
Furnace AA	Metals	90	110
	Tin	80	120
Cold Vapor AA	Mercury	80	120
Other	Cyanide	85	115

# Addicional Instrumental, QC Requirement

On a quarterly basis, instrument detection limits are determined for each ICP and AAS system used for the analyses of metals. This is accomplished by multiplying by three (3), the average of the standard deviations obtained on three (3) nonconsecutive days from the analysis of a standard solution of each AR 300573 analyte in reagent water. The concentration of each analyte in the standard solution is at 3-5 times the instrument detection limit and seven (7) concentrative measurements, per day, per analyte are required.

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On a quarterly basis, interelement and background correction factors are determined for ICP analysis using an Interference Check Sample. This measure determines the potential false analyte signals caused by the presence of high levels of certain common occurring elements found in environmental samples.

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## 7.4 Chain-of-Custody

The basic components for maintaining sample chain-of-custody are to ensure that the samples and aliquots/extracts are at all times either in the possession of the appropriate laboratory staff member or maintained in a secure area, and that adequate documentation accompanies the samples throughout the laboratory.

CompuChem® accomplishes these objectives through an elaborate document control system. This system includes procedures for documentation of the receipt of the sample into the laboratory using chain-of-custody records. These documents give information about the individuals taking the samples, the collection time, date, location and the type of analysis required. Though CompuChem® supplies instructions on the correct methods of sample collection, CompuChem's clients are responsible for sample collection. When the samples are received in the laboratory, these documents are signed by the receiving staff. The integrity of the samples within the laboratory are assured by the security of the facility itself. The building security is controlled by an electronic card entry system. The exterior doors and the doors of various controlled access areas within the building are equipped with card readers. Each member of the staff has an access card that is coded only for those areas where their job function requires access. For example, only those members of the staff who have responsibility for standard preparation are allowed access to that area. The system also maintains a record of the movements of the staff throughout the building. The original sample containers are kept in a locked refrigerator in in secure storage if refrigeration is not required) either during analys pending analysis. When the analysis is complete the final extracts for the

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extractable portions of the sample are kept in a locked freezer. These storage locations are the responsibility of the Sample Custodian.

A complete description of CompuChem's sample tracking procedures can be found in Appendix E. Chain-of-custody procedures are fully documented in the laboratory's Standard Operating Procedures Manual.

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#### 8.0 DATA PROCESSING

#### 8.1 Collection

Analytical data is generated from the GC/MS computer software, GC computer, ICP, Atomic Absorption Spectrophotometers, Technicon Autoanalyzer, and associated laboratory instrumentation. The outputs include identifications of compounds or elements, concentrations, retention times, and comparisons to standards. Outputs are in graphic form (chromatograms), bar graph (spectra) and printed tabular form. The outputs are in standard format specified for each analysis type and are monitored for consistency. If incomplete or incorrect output is received, corrective actions are taken according to procedures established for each type of analysis and consistent with the manufacturer's recommendations.

All outputs of each of the instruments are checked manually for each procedure (e.g., GC chromatographic peak area integration and calculations are checked manually for accuracy).

In the data review process (<u>see Validation</u>), the data produced are compared to information concerning the sample history, sample preparations. QC data, etc. to judge the validity of the results.

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tracking system forms, instrument logs, standard records, maintenance records, calibration records, and associated quality control. These sources are available for inspection during audits to determine the validity of data and many are also deliverable, depending on the client's needs.

A complete record of each sample's history must be available for documenting its progress through the laboratory from sample receipt to reporting. Document control and chain-of-custody requirements specified in those SOPs describe this documentation.

Data validation includes the use of dated and signed entries by analysts and supervisors on worksheets used for all samples; the use of sample tracking and numbering systems to logically follow the progress of samples through the laboratory, and the use of quality control criteria to accept or reject specific data.

Steps and checks used to validate precision and accuracy on the measured parameters and to support the represe 'ativeness, comparability and completeness of the work includ.

- Description of the calibration of methods and instruments;
- Description of routine instrument checks (noise levels, drift, linearity, etc);
- Documentation on traceability of instrument standards, samples and data;
- Description of applicable performance audits with appropriate and the materials; AR300578
- Description of the controls for interference contaminants in analytical methods (use of reference blanks and check si accuracy and precision);

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- Description of levels of routine maintenance to ensure analytical reliability; and
- Documentation on sample preservation and transport.

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### B.2 Validation

The analyst and supervisor review data to ensure the laboratory provides the following where appropriate:

- Calculates the recoveries of surrogate spikes;
- Verifies that there are no contaminants in associated blanks;
- Compares samples and duplicates for precision in data results;
- Reviews surrogate and spike recovery data to make sure they are within quality acceptance limits;
- Verifies calibration performance for acceptability;
- Reviews and verifies instrument tuning; and
- Reviews internal standard areas of response for acceptability.

Upon meeting all technical criteria; the sample folder is then reviewed by the Final Technical Review Staff to:

- Ensure surrogate recovery section has been completed and acceptance limits are not exceeded:
- Ensure that all analyte compounds have been properly recorded;
- Assure accuracy of calculations on compound quantitites; and
- Ensure confirmation by GC/MS has been performed and spectra are nesent.

The reviewer examines the entire sample folder to ensure that all data transcriptions and documentation included meet customer requirements. The Senior Technical Staff perform a final technical review to verify that the completed package conforms with all Quality Control criteria.

Upon completion of review, the customer report folders are forwarded to the Deliverables Department for mailing. AR300580

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# B.3 Report Storage

At every stage of data processing at which a permanent collection of data is stored, procedures are established to ensure data integrity and security. Specific QA project plans indicate how specific types of data are stored with respect to media, conditions, location, retention time, and access. The following chart indicates general guidelines as documented in the <u>SOP Manual</u>:

<u>Media</u>	Conditions	Location	Retention Time	Access
Hardcopy	locked warehouse	off-site	indefinitely (Comm) 120 days (EPA)	Sample Custodian or other designated personnel
Electronic	locked warehouse (environment	off-site	indefinitely	Facilities Manager or other designated personnel

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# 8.4 <u>Transcription</u>

All data transcriptions for final reports to commercial clients are performed by Report Integration Data Clerks and are reviewed by proof readers before reporting. For EPA-CLP reports, data transcriptions made by Report Integration Data Clerks are reviewed by the Final Technical Review staff.

Data trasnscription requirements vary but are monitored by the Supervisor of Report Integration in accordance with the various customer requirements for accuracy and legibility.

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### . 8.5 Data Reduction

Data reduction includes all processes that change either the values or number of data items. The original data set from which the new set is generated cannot be recovered from the new set.

Data reduction frequently includes computation of summary statistics.

Documentation of the calculation process is required. Frequently, a programmable calculator or computer will be used in this process. The documentation permits the reviewer to check the validity of the reduction process. All of the computer system-generated compound lists containing the reportable results include formulae used in the computation process.

It is CompuChem's policy to report results to two significant figures.

However, a minimum of one extra significant figure is carried through the calculations until the mathematical manipulations are complete, at which time normal rounding off processes are applied.

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#### 9.0 DATA QUALITY ASSESSMENT

#### 9.1 Introduction

<u>Precision</u>: The Laboratory objective for precision is to equal or exceed the precision demonstrated for the applied analytical methods on similar samples. Relative Percent Difference (RPD) criteria, published by the EPA as part of the EPA's Invitation for Bid (IFB) Contract Laboratory Program (CLP) for organic and inorganic analyses and those determined from laboratory performance data, are used to evaluate precision between matrix spike duplicates. The formula for determining Relative Percent Differences (RPD) is:

MS = spike recovery for matrix spike MSD = spike recovery for matrix spike duplicate

Accuracy: The Laboratory objective for accuracy is to equal or exceed the accuracy demonstrated for the applied analytical methods on similar samples. Percent Recovery Criteria, published by the EPA as part of the EPA's-IFB-CLP for organic and inorganic analyses, those published in the Federal Register (October 26, 1984), and those determined from laboratory performance data, are used to evaluate accuracy in matrix spike and blank spike Quality Control samples. The formula for determining accuracy is:

Concentration Measured X 100 = Percent Recovery Concentration Spiked

Representativeness: The representativeness of the data from the sampling sites depends on the sampling procedures. The representativeness of the analytical data is a function of the procedures used in processing the samples. The objective for representativeness is to provide data of the same high quality as other analyses of similar samples using the same methods during the same time

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period within the laboratory. Representativeness can be determined for this objective by a comparison of the quality control data for these samples against other data for similar samples analyzed at the same time.

Comparability: The results of analyses can be compared with other analyses by other laboratories because the objectives of the laboratory for comparability are: to demonstrate traceability of standards to NBS or EPA sources; to use standard methodology; to report results from similar matrices in consistent units; to apply appropriate levels of quality control within the context of the Laboratory Quality Assurance Program; and to participate in interlaboratory studies to document laboratory performance. By using traceable standards and standard methods, the analytical results can be compared to other laboratories operating similarly. The QA Program documents internal performance, and the interlaboratory studies document performance compared to other laboratories. Quarterly laboratory proficiency studies are instituted as a means of monitoring intralaboratory performance.

Completeness: CompuChem's objective for completeness is to be able to provide analyses for 100% of samples received intact and for which back-up sample is available should initial analysis not meet acceptance criteria. When requested, the completeness of an analysis can be documented by including in the report sufficient information to allow the data user to assess the quality of the results. The information delivered may include such items as: chromatograms, spectra, QC data, and summaries of results. Additional information, such as the Laboratory worksheets, notes, etc. are stored with the sample results in the Laboratory. The raw data (prior to data reduction) are archived in the laboratory whether or not the client requests results substantiation.

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## 9.2 Methods For Attaining Quality Control Requirements

The analytical and quality control requirements for each sample are achieved by means of our Computerized Laboratory Management System (CLMS). The System Analysis Codes are associated with specific Sample Preparation and Instrument Procedures and are dependent on sample matrix, fraction type, QC requirements, and detection limit requirements.

The Analysis Codes have associated with them Quality Control samples to be tripped automatically by the CLMS upon sample receipt. The particular types and frequencies of QC samples processed with a production sample are outlined in the USEPA Caucus Organics and Inorganics Protocols for the Contract Laboratory Program (CLP) 1985. Additional requirements are presented in other analytical method references (Federal Register, October, '84; customer specific QC sample requirements; EPA's SW-846 (Third Edition) manual; Project Specific and State certifying agency specific requirements). This will include, for instance, the application of blanks, duplicates and spikes at a frequency of one each for every batch of samples, or each type of matrix or 20 samples whichever, is more frequent, for the State of California. Following this section are tables of control limits for inorganic and organic QC requirements. Surrogate standards are used with each sample processed for organic analyses.

Blind samples are routinely sent to the laboratory for analysis. These may take the form of replicates as well as using external quality control samples. The samples are obtained from outside sources and contain known concentrations of specific compounds or are produced in the Standards Laboratory.

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#### Organic Analysis

Organics surrogate recoveries are used to determine whether the sample processing and analysis functions are in control. The pesticide surrogate, dibutylchlorendate, is presently used "for advisory purposes only" (although a minimum recovery of 10% is required); all other organic surrogates must be within the specified control limits for the sample fraction. Matrix spike control limits for organics samples associated with the EPA-CLP are also "for advisory purposes." Samples processed following procedures designated in the October, '84 Federal Register must meet acceptance criteria specified therein. The CLP methodologies require the calculation and documentation of Relative Percent Differences (RPDs) between recoveries of the matrix spike and matrix spike duplicate, although acceptance criteria have not been formally established. CompuChem<sup>2</sup> has adopted internal accuracy and precision criteria to be used as decision guidelines where the contract provides "advisory" criteria.

More than one-half of the QC spiking compounds must be recovered within acceptance criteria for each organic fraction. Similarly, more than one-half of the precision criteria (RPD) must be met per analytical fraction. If the criteria are not met, the matrix spike and matrix spike duplicate tests have to be repeated. For Federal Register requirements, full sample matrix spikes are performed for organic analyses. A blank spike is also processed with the sample spike. If all compounds in the sample spike are not recovered within acceptance criteria, the blank spike is analyzed. If neither QC sample meets criteria, the entire batch is reprocessed.

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#### <u>Inoganic Analysis</u>

Aside from the QC samples identified earlier and for which control limit tables are presented following this section, two other QC measures, dealing with ICP analyses are employed.

At the beginning and end of each analysis shift, an ICP Interference Check Sample is analyzed. This analysis verifies interelement and background correction factors since it assesses analytes of interest in the presence of high concentration levels of other elements. Control limits for this test are presented following this section.

Additionally, for each batch of samples processed, an ICP Serial Dilution Analysis is performed. If an analyte is present at a sufficiently high enough level (minimally a factor of 10 above the instrumental detection limit), an analysis of a 1:4 dilution should agree with 10% of the original determination. If not within that limit, a chemcial or physical interference effect is likely, and the associated data would be qualified.

Minimum criteria for the evaluation and frequency of method blanks are addressed in their applicable method references. The Quality Assurance Department routinely audits method blank data to ensure that criteria are being adhered to and that potential sources of contamination are identified before samples are affected. In addition, numerous Quality Control samples are introduced regularly into the system to monitor the cleanliness of the glassware preparation operation, volatile instrumentation, volatile sample bottle storage facility, inorganics sample bottle preparation, and sample storage are facility, inorganics sample bottle preparation, and sample storage facility assurance SOPs in the Environmental SOP Manual. Criteria of acceptance are outlined in that document

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The management and staff of CompuChem® make every attempt to generate data of the highest quality possible and will continue to apply state-of-the-art analytical methodologies to ensure that our data continues to be of the best quality available anywhere.

CompuChem® makes every attempt to produce and deliver analytical data which has been demonstrated to meet contract—, method—, or client—required quality control acceptance criteria. Should anomalies occur in the processing and/or analysis of samples which affect that objective, Quality Assurance or Laboratory Notices are typically generated and delivered with the data results to serve as qualifiers.

As described earlier, in this section, precision and accuracy acceptance limits for CLP (Contract Laboratory Program) organic and inorganic analyses are contract-mandated. CompuChem also offers a variety of analytical services using Federal Register methodologies, and of course, the QC requirements for accuracy and precison are method-mandated. In the October 31, 1984 F.R., it is recommended that the laboratory periodically update these control limits based on historical data. It is CompuChem's intent to do so once a database of sufficient size is generated.

Control limits will be based on the following formulae:

LCL = x - 3S

UCL = x + 3S, where

LCL = Lower Control Limit

UCL = Upper Control Limit

X = Mean Percent Recovery

SD = Standard Deviation

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All QC sample results are tabulated immediately following analysis and compared to the contract-mandated, method-mandated, or client-mandated control limits for precision and accuracy. Out-of-control results are cause for immediate re-extraction and/or re-analysis. No outlying data are ever released until the laboratory has verified that unacceptable results are attributable to the sample matrix.

The laboratory is currently developing the software necessary to plot control charts for each sample matrix, concentration-level (Low/Medium Level), and sample type (acid, volatile, etc.). For all CLP analyses, precision and accuracy data are required to be tabulated and reported on the "MS/MSD Form III". These data are then statistically analyzed by the USEPA (EMSL-Las Vegas), and presented periodically to all CLP labs in "Spike-Exceptions Reports." In this way, both intra-lab and inter-lab trends in QC results can be observed.

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# COMMERCIAL ORGANIC AND EPA - CLP CONTRACT REQUIRED SURROGATE SPIKE CONTROL LIMITS\*

Volatile Surrogates	Solid	Liquid
D4-1,2-Dichloroethane	(70-121)	(76-114)
4-Bromof luorobenzene	(74-121)	(86-115)
D8-Toluene	(81-117)	(88-110)
Base/Neutral Surrogates	Solid	Liquid
D5-Nitrobenzene	(23-120)	(35-114)
D10-Pyrene	(17-125)**	(40-130)**
D14-Terpheny1	(18-137)	(33-141)
2-Fluorobiphenyl	(30-115)	(43-116)
Acid Surrogates	Solid	<u>Liquid</u>
2-Fluorophenol .	(25-121)	(21-100)
2,4,6-Tribromophenol	(19-122)	(10-123)
D5-Phenol	(24-113)	(10-94)
Pesticide Surrogate	<u>Solia</u>	Liquid
Dibutylchlorendate (DBC)	(20-150)***	(24-154)***
Herbicide Surrogate	<u>Solid</u>	<u>Liquid</u>
2,4-DB	(16-124)***	(28-104)***

<sup>#</sup> as noted in IFB (WA-85J 680/664, 7-85) and subject to modification based on data supplied in the CLP

<sup>\*\*</sup> laboratory optional surrogate only; no action limits at this time \*\*\* advisory surrogate; minimum 10% recovery used as action limit

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EPA-CLP MATRIX SPIKE RECOVERY LIMITS\*

Fraction	Matrix Spike Compound	Water*	Soil/Sediment
<b>Y</b> DA	1,1-Dichloroethene	67-145	59-172
<b>Y</b> OA	Trich loroethene	71-120	<b>62-137</b>
<b>Y</b> OA	Ch lorobenzene	<b>75-13</b> 0	<b>6</b> 0 <b>-1</b> 33
<b>V</b> OA	Toluene	<b>76-</b> 125	<b>59-1</b> 39
<b>V</b> DA	Benzene	<b>7</b> 6-127	<b>6</b> 6-142
BN	1,2,4-Trichlorobenzene	<b>39-9</b> 8	<b>3</b> 8-107
BN	Acenaphthene	46-118	31-137
BN	2,4-Dinitrotoluene	24-96	28-89
BN	Di-n-Butyl phthalate	11-117	29-135
BN	Pyrene	26-127	<b>35-14</b> 2
BN	N-Nitroso-Di-n-Propylamine	41-116	41-126
BN	1,4-Dichlorobenzene	36-97	28-104
Acid	Pentachlorophenol .	<b>9-1</b> 03	<b>17-</b> 109
Acid	Phenol	12-89	26-90
Acid	2-Chlorophenol	27-123	<b>25-1</b> 02
Acid	4-Chloro-3-Methylphenol	23-97	26-103
Acid	4-Nitrophenol	10-80	11-114
Pest.	Lindane	56-123	46-127
Pest.	Heptach lor	40-131	<b>35-13</b> 0
Pest.	Aldrin	40-120	<b>34-1</b> 32
Pest.	Dieldrin	52-126	31-134
Pest.	Endrin	56-121	7 139
Pest.	4,4'-DDT	38-127	≟134

<sup>\*</sup>These limits are for <u>advisory purposes only</u> (as noted in WA-8SJ680/664, 7-85). They are not to be used to determine if a sample should be reanalyzed. When sufficient multi-lab data are available, standard limits will be calculated.

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# WATER AND WASTEWATER QC ACCEPTANCE CRITERIA-METHOD 608.

# Federal Register, October 26, 1984

<u>Parameter</u>				 Percent Recovery
Aldrin				42 - 122
a lpha-BHC			•	37 - 134
beta-BHC				17 - 147
delta-BHC				19 - 140
gamma-BHC	(Lindane)			32 - 127
Chlordane				_ 45 - 119
4,4'-DDD				31 - 141
4,4'-DDE	-			 30 - 145
4,4'-DDT				<b>25 - 16</b> 0
Dieldrin				36 - 146
_Endosulfan		•		 45 - 153
Endosulfan				D - <b>2</b> 02
Endosulfan	Sulfate			26 <b>- 1</b> 44
Endrin				30 - 147
Heptach lor				 34 - 111
Heptach lor	epoxide			37 - 142
Toxaphene	- •			41 - 126
PCB-1016				50 - 114
PCB-1221			,	15 <b>- 1</b> 78
PCB-1232				10 - 215
PCB-1242				 39 <b>- 1</b> 50
PCB-1248				38 - 158
PCB-1254				29 - 131
PCB-1260				8 - 127

# WATER AND WASTEWATER QC ACCEPTANCE CRITERIA

#### **HERBICIDES\***

2,4-D	38 - 15	2
2,4,5-TP	35 - 14	2
2,4,5-T	38 - 14	1

<sup>\*</sup>Advisory use only; minimum 10% recovery used for action limits.AR300593

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# WATER AND WASTEWATER QC ACCEPTANCE CRITERIA-METHOD 624

# Federal Register, October 26, 1984

Parameter	-	Percent Recovery
Benzene		37 - 151
Bromodichloromethane		<b>35 - 15</b> 5
Bromoform		45 - 169
Bromomethane		_ D - 242
Carbon Tetrachloride		70 - 140
Ch lorobenzene		<b>37 - 16</b> 0
Ch loroethane		14 - 230
2-Chlorethylvinyl ether		D - 305
Chloroform		51 <b>- 1</b> 38
Ch loromethane		D - 273
Dibromochloromethane		53 - 149
1,1-Dichloroethane		<b>59 - 1</b> 55
1,2-Dichloroethane		49 - 155
1,1-Dichlorothene		D - 234
trans-1,2-Dichloroethene		54 - 156
1,2-Dichloropropane		D - 210
cis,1,3-Dichloropropene		D - 227
trans-1,3-Dichloropropene		17 - 183
Ethyl benzene		37 - 162
Methylene chloride		D - 221
1,1,2,2-Tetrachloroethane		46 - 157
Tetrachloroethene		64 - 148
Toluene		47 - 162
1,1,1-Trichloroethane	-	52 - 162
1,1,2-Trichloroethane		<b>52 - 150</b>
Trichloroethane		71 - 157
Vinyl chloride		D - 251
Acrolein		D - 150
Acrylonitrile		<b>D - 1</b> 50

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# WATER AND WASTEWATER QC ACCEPTANCE CRITERIA-METHOD 625

# Federal Register, October 26, 1984

<u>Parameter</u>		Percent Recove	<u>ry</u>
Acenaphthene		47 - 145	
Acenaphthylene		33 - 145	
Anthracene		27 - 133	
Benzo(a)anthracene		<b>33 - 14</b> 3	
Benzo(a)anthracene Benzo(b)fluoranthene Benzo(k)fluoranthene		24 - 159	
Benzo(k)fluoranthene		24 - 159 11 - 162 17 - 163 D - 219	
Benzo(a)pyrene		17 - 163	
Benzo(ghi)perylene		D - 219	
Benzyl butyl phthalate		D - 152	
Bis(2-Choroethyl)ether		12 - 158	
Bis (2-chloroethoxy)methane		33 - 184	
Bis(2-chloroisopropyl)ether		30 <b>- 10</b> 0	
Bis (2-ethylhexyl)phthalate		<b>8 - 15</b> 8	
4-Bromophenyl phenyl ether		<b>53 - 127</b>	
2-Chloronaphthalene		60 <b>- 1</b> 18	•
4-Chlorophenyl phenyl ether		<b>25 - 15</b> 8	
Chrysene		<b>17 - 16</b> 8	
Dibenzo(a,h)anthracene		D - 227	
Di-n-butyl phthalate		1 - 118	
1,2-Dich lorobenzene	• •	<b>3</b> 2 - 129	
1,3-Dich lorobenzene		D - 172	
1,4-Dichlorobenzene	•	20 - 124	
3,3'-Dichlorobenzidine		D - 262	
Diethyl phthalate		D - 114	
Dimethyl phthalate	•	D - 112	
2,4-Dinitroto luene	· · · · · · · · · · · · · · · · · · ·	39 - 139	
2,6-Dinitrotoluene	<del>-</del>	50 - 158	
Di-n-octylphthalate		4 - 146	
Fluoranthene		26 - 137	
Fluorene	· · · · · · · · · · · · · · · · · · ·	59 - 121	
Hexach Torobenzene		D - 152	
Hexachlorobutadiene		24 - 116	
Hexach Toroethane		- 40 - 113	
Indeno(1,2,3-cd)pyrene		D - 171	
Isophorone		21 - 196	
Naphtha lene		21 - 133	
Nitrobenzene		35 <b>- 18</b> 0	6 D O D -
N-Nitrosodi-n-propylamine		D - 230	AR300595
Phenanthrene		54 - 120	·
Pyrene		<b>52 - 115</b>	

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1,2,4-Trichlorobenzene	44 - 142
4-Chloro-3-methylphenol	22 - 147
2-Ch lorophenol	23 - 134
2,4-Dichlorophenol	<b>39 - 13</b> 5
2,4-Dimethylphenol	<b>32 - 1</b> 19
2,4-Dinitrophenol	อ - 191
2-Methyl-4,6-dinitrophenol	D - 181
2-Nitrophenol	<b>29 - 18</b> 2
4-Nitrophenol	D - 132
Pentach lorophenol	14 - 176
Phenol	5 - 112
2,4,6-Trichlorophenol	37 - 144

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# SOLID QC ACCEPTANCE CRITERIA

Parameter	Concentration of Spike Added (ug)	Percent Recovery
1,1-Dichloroethane Trichloroethene Chlorobenzene Toluene Benzene	0.20 0.20 0.20 0.20 0.20	59 - 172 62 - 137 60 - 133 59 - 139 66 - 142
1,2,4-Trichlorobenzene Acenaphthene 2,4-Dinitrotoluene Di-n-Butyl phthalate Pyrene N-Nitroso-Di-n-Propylamine 1,4-Dichlorobenzene	100 100 100 100 100 100	38 - 107 31 - 137 28 - 89 29 - 135 35 - 142 41 - 126 28 - 104
Pentachlorophenol Phenol 2-Chlorophenol 4-Chloro-3-Methylphenol 4-Nitrophenol	100 100 100 100 100	17 - 109 26 - 90 25 - 102 26 - 103 11 - 114
Lindane Heptachlor Aldrin Dieldrin Endrin 4,4'-DDT	0.20 0.20 0.20 0.50 0.50 0.50	46 - 127 35 - 130 34 - 132 31 - 134 42 - 139 23 - 134
2,4-D 2,4,5-TP 2,4,5-T	40 10 10	56 - 160 61 - 113 63 - 109

<sup>\*</sup> Soil-modified methods 624, 625, 608, and 615, based on acceptance criteria noted in IFB-WABSJ680/664, 7-85, except herbicide.

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#### 10.0 Corrective Action

#### 10.1 Introduction

Generally, there are two types of corrective actions that may be required when data quality falls below specified limits. The first type, and the simplest to implement and document, is corrective action required because <u>routine</u> data quality assessments are out-of-control. Surrogate and spike standard recoveries, relative percent differences between duplicates, internal standard response variations, and unacceptable blank contamination are some of these assessments in the first category. These are all performed on a sample-by-sample and/or batch basis, and corrective action is limited to evaluating the data with respect to SOP criteria, and accepting or rejecting the sample/batch. The decision that is made is clearly indicated on analytical worksheets, and unless a trend is observed during the course of data validation, additional corrective action or documentation is not necessary.

The second type of corrective action is that required when other, more global QC/QA assessments, are made. The assessments might typically indicate systematic deficiences or those affecting data useability for more than one batch (i.e., glassware contamination checks, standards preparation errors, etc.). In most cases, assessments of this nature are made by reviewing peripheral QC/QA documentation, observing procedures for comparison with SOPs or GLPs, or receiving feedback from data reviewers, management or those external to the organization (clients, auditors).

The following sections describe the QA reporting and feedback channels designed to ensure that early and effective corrective action is taken in such instances.

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In many cases, depending on the nature of the deficiency and the urgency for remedial action, a Corrective Action Report (following this section) will be completed. The report serves to document the deficiency, the required corrective action, and accountability for the action.

For observations made over longer periods of time, the QA Department issues formal summary reports to management on a monthly or quarterly basis. Following is a brief discussion of the types of reports issued to management to assess the overall effectiveness of the QA Program and to reinforce the application of Good Laboratory Practices\_(GLPs).

# CORRECTIVE ACTION REPORT

DATE:	
PROBLEM / DEFICIENCY:	
• · · · · · · · · · · · · · · · · · · ·	·
THENTYFIED BY	
REFERRED TO:	
REPERRED 10:	(Qn)
CORRECTIVE ACTION TO BE TAKEN:	AREET DATE:
	4
FOLLOW-UP AUDIT FINDINGS:	•
RESOLVED? DATE:	AR300600
SOP REQUIRED TO BE WRITTEN/MODIFIED? YES [ ] NO	[ ] TARGET DATE:
This form to be filed with the Quality Assurance Cle	rk for permanent record

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## 10.2 Routine QC Check Reports

The following routine quality control checks (also discussed in section 9.2 of the QA Plan) are performed to verify that samples are not contaminated during transportation, preparation, analysis or storage, and that standards prepared internally are traceable to certified sources.

- -- Vendor-Supplied Glassware Checks
- -- Glassware Decontamination Checks
- -- Water Purification Systems Checks
- --- Glassware Storage Cabinet Checks
- -- Refrigerated Storage Systems Checks
- -- Reagent Purity Checks
- -- Standards Prepartion and Traceability Checks

The criteria for these QC checks and corrective action steps are detailed in the QA SOP Manual. Results are tabulated and/or plotted on control charts, and records reviewed by the QA staff. A series of quarterly reports to management summarize this information and the status of these programs.

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## 10.3 Monthly QA Activity Reports

These reports are produced by all members of the QA staff, and summarize key QA activities during the previous month. The reports are distributed to the Director of QA, and are provided as an attachment and referenced in the Director's report to the CEO, the Executive Staff and senior laboratory management.

Included in these reports is a summary of significant quality problems observed during the period, and the corrective actions taken to remove deficiencies. The report stresses proactive measures that are being taken to improve quality or ensure compliance with QA program requirements.

Laboratory management uses the report to quantitatively measure monthly performance in terms of the number of samples processed, the frequency of repeated sample analyses due to unacceptable QC performance, and the cause of the unacceptable performance. These data are all presented in tables, Pareto control charts or attribute control charts, based on the characterization of each analysis in the Computerized Laboratory Management System (CLMS) using a system of analytical "condition codes."

The Condition Code System is used to monitor sources of data failures.

Condition code definitions are provided in an SOP to data generators and reviewers who are responsible for assigning the appropriate code to each analysis (see Appendix D). Each two-letter code is used to characterize the cause of a sample failure or the final status of the data package prior to release to the client.

AR300602

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Various computer programs may be used to sort condition code data according to sample matrix and method. This system is used to pinpoint sources of error, provide feedback to management, reinforce good laboratory practices, and document laboratory performance over time. The QA staff also note in the Monthly QA Activities Report any corrective actions taken or necessary procedural changes, based on the application of condition codes.

Other items included in this report are:

- -- Summary of any changes in certification/accreditation status
- -- Involvements in resolution of quality issues with clients or agencies
- -- QA organizational changes
- -- Notice of the distribution of revised documents controlled by the QA Department (i.e., SOPs, QA Plan)
- -- Training and safety issues, if not already covered in audit reports during the period
- -- Performance of subcontractor laboratories (also communicated in separate, detailed subcontractor audit report to management)
- -- Positive feedback for acceptable performance on interlaboratory or intralaboratory tests or successful completion of audits.

AR300603

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## 10.4 <u>Laboratory Performance Reports</u>

This quarterly report presents a statistical and graphical summary of the laboratory's performance on batch-associated quality control samples analyzed over the period. Included are tables, Shewhart control charts and I-charts (for individual data points) for all surrogate and spike standard recoveries. Additionally, a monthly report to the Director of QA presents control charts and tables for all Laboratory Control Sample (Blank Spike) and Blank recoveries. The charts and tables are used primarily to document historical performance, update recovery control limits, and monitor long-range trends that might not be apparent to data reviewers evaluating data on a sample/batch basis.

AR300604

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## 10.5 Laboratory Audit Reports

Quarterly audit reports are written by a member of the QA staff and distributed to management, and summarize the results of internal laboratory Performance Audits, Systems Audits and Security/Access Audits. When external auditors are involved in Performance or System Audits, a report is written within the next week by the QA staff member coordinating the audit. The report, summarizing audit results as discussed in the debriefing as well as other observations, is distributed to the CEO and senior lab management. The report includes corrective actions required as a result of the audit, and a schedule for implementation. A follow-up audit, usually within three weeks of the distribution of this report, is conducted to verify that corrective actions have been implemented.

## Performance Audits

Performance Audits are checks made by a QA staff member or other independent auditors to evaluate the quality of the data produced by the analytical system. These audits are performed independent of and in addition to routine quality control checks, and reflect as closely as possible lab performance under normal operating conditions.

These audits involve the review of approximately 10% of all analytical data reports generated by the lab for calculation and data validation procedures, and overall data quality. Errors observed during the audit are characterized as "critical" or "correctable" and tabulated. If necessary, based on audit findings, an amended data report may be sent to the customer. Following the \$605 section is a copy of the QA Audit Summary used by auditors to tabulate the data

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for summary into the Quarterly Performance Audit report. A thorough discussion of these audits is included in the QA SOPs. The reports are used by laboratory managers to provide feedback to staff members and establish goals for improved performance.

A number in interlaboratory and intralaboratory tests are conducted routinely at CompuChem®, and the results are included in individual Performance Audit reports specific to each test. When new methods are available to the laboratory or new personnel are being trained, Laboratory Proficiency Tests are performed. These tests consist of quadruplicate blank spikes, containing a full complement of tests parameters to be analyzed by the method. The replicate results are analyzed by a QA staff member, who generates a summary report to the Director of QA. This report includes the standard deviation and mean recovery for each of the replicate parameters, and the data are used to statistically validate method and/or personnel proficiency. For a thorough discussion of the method validation procedures used, refer to Appendix A of the QA Plan.

On a quarerly basis, blind intralaboratory check samples are introduced into the system by the QA Department. Parameters and methods are chosen for these studies based upon independent (interlaboratory) tests from certifying agencies (including the U.S. EPA and various state agencies), Laboratory Proficiency Test results, Method Validation studies, or results from routine batch-related QC samples. The existence of these check samples in the system is known only to those personnel involved in preparing the samples and scheduling the analytical requirements into the CLMS. A thorough report, detailing the entire data generation and support functions, is completed by the QA staff ARGORGE 6

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the	Director	of Q	A before	distr	ibution	to	the	CEO	and	senior	laboratory
man:	agement.		-·· ·								
### T	agement.								-		

CompuChem® also participates in a number of external, interlaboratory performance studies. These are required as part of various agencies' certification/accreditation programs. As a member of the USEPA's Contract Laboratory Program (CLP), the laboratory is required to successfully analyze quarterly, blind proficiency samples for both organic and inorganic parameters. The CLP program also requires an annual on-site inspection by principals from the USEPA (and their contracted agents). These audits generally follow the same format described below, Systems Audits.

CompuChem® also participates in a number of state certification programs, including those for North Carolina, New Jersey, New York and Florida. All of these programs require the laboratory to submit to annual on-site inspections in order to maintain certification to perform testing on samples originating in the state. All states also require successful performance on interlaboratory check samples, submitted at least annually, though some reciprocity with the two NC programs (one for drinking water and one for wastewater certification) and USEPA-CLP is allowed under certain circumstances.

Several states utilize the laboratory's performance on the annual Water Supply (WS) and Water Pollution (WP) proficiency testing series, orginating out of the EPA Environmental Monitoring and Support Laboratory's performance on all interlaboratory and intralaboratory check samples, tabulated by parameter and method, so negative performance trends can be readily pinpointed.  $^{AP300607}$ 

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#### System Audits

A System Audit is an on-site inspection and review of the QA Program for the total laboratory. While Performance Audits are a quantitative appraisal, System Audits are for the most part qualitative in nature. The System Audit may be either scheduled or unannounced before it is conducted, but occurs routinely on at least a quarterly basis. The auditor reviews the laboratories' SOPs to verify compliance with procedures and activities actually in place. Personnel and facilities are also evaluated during the System Audit. The auditor is required to investigate anything which seems in conflict with the QA Plan, the laboratory or QA SOPs, or Good Laboratory Practices.

If deficiencies are observed during a Performance Audit, and if deemed necessary, the QA Department initiates a System Audit. The audit emphasizes the actions necessary to correct deficiencies noted in the Performance Audit. A Corrective Action Report is completed, detailing all remedial actions taken, and reviewed by the Director of QA. The report must indicate the proposed implementation date and the individual(s) responsible for the action.

Many of the objectives of a routine System Audit are similar to those a client or independent auditor would hope to accomplish during an On-Site Laboratory Evaluation and Data Audit. These goals include ensuring the following:

- 1. The quality control, including necessary corrective actions , are being applied and a management of the second and applied applied
- Adequate facilities and equipment are available to perform the client's required scope-of-work

  AR300608
- 3. The personnel are qualified to perform the assigned tasks
- 4. Complete documentation is available, including sample chain-of-custody

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- 5. Proper analytical methodology is being applied
- 6. Acceptable data handling techniques are being used
- 7. Corrective actions identified in any previous on-site visits have been implemented, and
- 8. The Laboratory Management continues to demonstrate a commitment to quality.

These objectives may be documented by completing an EPA-approved Laboratory

Evaluation Checklist. In response to System Audits, any corrective actions
taken are noted with reference to the auditor's deficiency report and the lab's

Standard Operating Procedures.

QUALITY ASSU

				2	REJECT L	LOCATION/CAUSE	I/CAUSE			HIMMARY	
	SAMPLE NUMBER	FRACTION						COMMENTS	Failurefailure	crit:	status
,		7						•			
•	2										
•	c							٠			
	•							•			
•	**										
•	•										
	,							•			
•	•										
•	G										
, '	10										
. '	1.1										
•	12										
-	13						,				
-	**										
'	16										
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'	1.1										
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•	10										
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•	20							-1			
								* F. Planes		DATE:	

#### QA AUDIT CODES

# Missing/Incorrect:

CAM/I Calculations missing/incorrect CCM/I Condition code missing/incorrect DFM/I Data footnote missing/incorrect CFM/I Correction factor missing/incorrect DWI Dry weight/percent moisture incorrect Filename incorrect FNI FFM/I Form 4 missing/incorrect LSM/I Library search missing/incorrect QNM/I QA Notice missing/incorrect RRM/I Reportable run missing/incorrect SPM/I Spectrum missing/incorrect SRM/I Sample receiving information missing/incorrect Surrogate Summary Form missing/incorrect SSM/I STM/I Standard package missing/incorrect JFM/I Tuning Form missing/incorrect UNM/I Units missing/incorrect WSM/I Worksheet missing/incorrect/incomplete OAM/I OADS missing/incorrect/incomplete

#### Qualitative/Quantitative Errors:

HNR Hit not reported, but should have been HRE Hit reported in error, should not have been reported HAI Hit amount reported incorrectly CFN Correction factor not applied to hit SFI Significant figures (or rounding off) incorrect TRE Transcription error

## Miscellaneous Errors:

ISF Internal standard area monitor indicates failure ODI OWA date or time incorrect
RNL RIC not labeled
SOL Surrogate(s) actually outside limites
WOU Whiteout used on documents (deliverables)
NSO Not signed off
CNI Change not initialed

# Condition Code Applications:

		•
<b>C</b> S	Carryover suspected	
CT	Contamination evident	AR300611
ŔŰ	Repeated unnecessarily	HILOGOGII
SF	Spikes failed	
UN	Unacceptable, not needed	•

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#### 10.6 Subcontracted Services

Subcontract services are regulated to comply with the requirements of the Quality Assurance Progam. The Marketing Department establishes, with input from the laboratory, when subcontract requirements are needed. The QA Department verifies that the subcontractor complies with the methods written in their referenced SOPs. This is accomplished by an on-site inspection of the subcontractor facility. The same criteria and objectives used during an internal Systems Audit are used for the subcontractor audit. Prior to the approval of a laboratory for its analytical services, blind PC samples are submitted and must be successfully completed as part of their performance audit.

The Director of QA has final authority over the approval of all subcontractor services. CompuChem's clients are notified whenever a subcontractor is to provide analytical services. Subcontractors are not used when specifically restricted by a client's QA Project Plan.

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# 11.0 IMPLEMENTATION

The implementation of this QA Plan is complete upon the distribution of this document to laboratory managers and other personnel.

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APPENDIX A

METHOD VALIDATION STUDIES

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# General Approach for the Validation of Analytical Methods by the Laboratory

#### Introduction

Historically our laboratory has determined the viability of published procedures by performing lab proficiency tests; i.e.-full analyte spikes are added to quadruplicate aliquots of laboratory pure water or "blank" soil, the samples are prepared/extracted and then analyzed by the appropriate instrumentation. The instrumentation would include GC/MS, GC, with appropriate detectors, and in the inorganic area, ICP/AAS/cold vapor AAS.

The laboratory proficiency testing program has been beneficial in demonstrating to ourselves and to interested clients that our applications of specified analytical methodologies are capable of producing acceptable data. The acceptable data is further characterized with statements of accuracy and precision; mean percent recoveries and standard deviations, respectively.

A necessary complement to the laboratory proficiency tests would be a formalized method detection limit study.

Before describing the rudiments of a recommended \*Generic Method Validation Study,\* certain definitions of terms are required. John K. Taylor (1) of NBS presents the following definitions of the hierarchy of methodology; from the general to the specific:

- (1) A Technique is a scientific principle, useful for providing compositional information.
- (2) A Method is a distinct adaptation of a technique for a selected measurement purpose.
- (3) A Procedure is composed of the written directions necessary to utilize a method.
- (4) A Protocol is the most specific name for a method and contains a set of definitive directions that must be followed, without exception, in order that the results be accepted for a given purpose.

Additionally, in an article entitled "Principles of Environmental Analysis" (2), a distinction is made between verification and validation:

- (1) Verification is a general process used to decide the capability of Commetted for producing accurate and reliable results.
- (2) Validation is an experimental process by other laboratories (internal or exreference materials in order to evaluate the

nal corroboration the use of methodology 14

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Our laboratory is embarking on a second generation of testing requirements which will serve to formally "validate" the "methods" we employ for new product offerings. The "Generic Method Validation Study" will serve to supply the data needed to satisfy ourselves and our clients that the laboratory's approach is sound. The impetus will be on the individual laboratories to prepare the specific experimental design, based on the method being validated. Additionally, the individuals actually performing the sample preparation, instrument calibration, analysis and data reduction processes will be required to utilize laboratory notebooks. The purpose of the laboratory notebooks is fourfold:

- (1) To record observations concerning problems encountered in applying the experimental design as written,
- (2) To note recommendations which may serve to eliminate the problems experienced,
- (3) To serve, with the experimental design, as a basis for the Standard Operating Procedures (SOPs) which will subsequently be required, and
- (4) To provide a basis for the preparation of an "Equivalency" petition to be submitted to the EPA.

(Note: as indicated above under the definitions of the hierarchy of methodology, a Procedure should not be able to be written until a Method has been utilized; i.e. - until the laboratory testing of the Method is accomplished and the details of the tests; problems/observations/recommendations, as written in laboratory notebooks, are evaluated).

In validating a method, the kinds of samples (matrices) to be processed should be clearly described. As a result of the validation process, statements of precision and accuracy will be generated. It should be realized that these data serve only as an estimate of the typical performance expected.

In being able to judge the suitability of a method, other factors have to be considered: sensitivity to interferences, limits of detection and useful range of measurement (1).

Interferences may come from two sources: those that are inherent in the matrix and laboratory artifacts, introduced during the sample processing.

By running appropriate method blanks and/or unspiked controls, the interferences can be characterized.

The concepts of detection limits and quantitation limits require elucidation - (2).

ARRODEIC

The Limit of Detection (LOD) is defined as the lowest concentration level that can be determined to be statistically different from a blank.

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- The Method Detection Limit (MDL) is the lowest concentration of analyte that a method can detect reliably in a sample or blank.

- The Instrument Detection Limit (ID) is defined as the smallest signal above background noise that an instrument is able to detect reliably.
- The Limit of Quantitation (LOQ) is defined as the level above which a specified degree of confidence may be otained for the quantitative results.

Our lab has historically used published detection limits or contract required detection limits. In situations where we are validating methods for which there are no detection limits (method or contractually defined), it will be our responsibility to correctly develop detection limits. It is important to understand the concepts since the reportable results will fall into different regions of reliability.

The "General Method Validation Study" write-up which follows is written for those methods which have already been written; the data produced from the study presents our application of the method. If methods are truly developed by us, the number of samples will be required to increase since statistical considerations suggest that at least six degrees of freedom (ordinarily seven measurements) should be involved at each decision point.

Classical validation processes involve the use of standard reference materials (SRM) after generating preliminary data. This approach is more viable when the SRM are similar in all respects to the test samples. The use of SRM may be appropriate as a final validation step if the number and type of analytes, and the matrix is the same. However, since many of the methods to be validated must encompass a variety of matrices and a cross-section of analytes, SRM may not be available. This will not preclude the use of those materials as part of a QA program to periodically insure us that our analytical systems are under control.

#### Generic Method Validation Procedure

The procedure being presented here is applicable for the GC/MS, GC, and inorganics laboratories. The purpose of the testing program is to generate precision, accuracy and recovery data on an aqueous and solid matrix, spiked with analytes of interest at one specified concentration. It should be used to gain experience and to demonstrate our laboratory's capabilities in applying procedures which have already been written; e.g. SW 846 Methods, Method 601, 602, etc. If our laboratory is truly developing a new method, another testing scheme would be applied.

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Validation of an organic method using only water and sand matrices is judged to be suitable only for those instances where one or more surrogates can be used to monitor the effectiveness of the method in more complex matrices. For those organic methods where surrogates are not employed, testing additional matrices, e.g. - clay, planter's mix should be incorporated into the validation processing.

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A method validation study requires that laboratory notebooks be utilized in order to record any observations/problems encountered. Realistically, our SOPs should not be written until we have experience in applying the method being evaluated.

The conduct of, and the results from, each step are to be documented in laboratory notebooks. The notebooks should also serve to record any recommendations which can be made concerning a better application of the sample processing, analysis, or data evaluation steps. The steps to be taken in this validation process are as follows:

#### 1. Desk Top Review

The method as written is read by a chemist familiar with extraction/work up procedures and the instrumental detection systems required. During this reveiw, the chemist will particularly look for:

- A. Safety hazards.
- B. Applicability of available instrumental systems.
- C. New equipment/systems required that are not available.
- D. Discrepancies in the write up which do not appear to make sense from a chemical analysis standpoint. Exceptions to the write up need to be clearly identified.
- E. QA/QC requirements
- 2. Preparation of Lab Plan

The lab plan is essentially, the testing approach to be taken and includes the proposed members of the "team" conducting the study and the specific exceptions, if any, to be taken from the method as written. The lab plan will be presented for approval to a review committee consisiting of Lee Myers, Chuck Bannerman, Ross Robeson and Bob Meierer.

# 3. Preparation of Draft Method

The draft method will be written. Use of a xerographic copy from a standard manual is acceptable.

As an appendix to this draft method, the laboratory manager or project manager shall present the compounds to be spiked into the factors tested. The analytes composing the spiking solutions should be all those (organic or inorganic) for which the method is being validated.

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Subsequent laboratory proficiency tests or standard reference materials, will be used on a routine basis to provide additional data on our application of the procedure.

#### 4. Laboratory Analysis

The matrices being evaluated are clean sand and laboratory pure water.

Method blanks consisting of aliquots of the sand and water are required.

Surrogate(s) are required for all organic procedures being evaluated.

When spiking these test samples, a minimum of one hour should elapse after spiking and thoroughly mixing and before the sample preparation process. Recommendations for modifications to volatile spiking requirements will be considered.

The spiked matrices shall be prepared and analyzed using the method write-up prepared under item 3 above. If our method differs from the published method, both must be run.

The spiking level to be analyzed in quadruplicate is:

An exact spiking level cannot be specified because the overall method recoverability is not known. Approximations of the recoverability can be made and used to prepare the spiking level. Alternatively, preliminary data points can be obtained by generating some recovery data on one or more spikes, using an estimate of a mid-level concentration.

#### 5. Detection Limit Run

After the data from section 4 (Laboratory Analysis above) is obtained, a formalized Method Detection Limit Study should be performed following the design specified by the EPA (for both water and sand matrices) in October 26, 1984 Federal Register.

## 6. Summary Report Requirement

The written report, documenting the experimented effort, will be submitted to the Vice President, Laboratory Operations, for review. This report will include as a minimum:

a. Safety requirements for routine operation of the method 310 06 19 laboratory.

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b. A full description of the method including all procedures and equipment used. This description must highlight deviations from the method as written in the applicable government regulation or manual (e.g. SW-846 Manual, etc.).

- c. A description of the matrices tested.
- d. A comparison of results obtained with our method if different from the published method. Data should be tabulated to present actual results per test sample per compound/element and the mean recoveries and % RSD data. These reports should be as detailed as possible since they will serve a threefold purpose:
  - They will serve as the basis for the preparation of written SOPs,
  - They will be used in marketing efforts for new product offerings and will clearly demonstrate the extra effort which CompuChem takes in providing analytical data of the highest quality, and
  - Serve as the basis for documenting requests for equivalency of CompuChem methods to EPA published methods (if necessary).
- e. An assessment of any factors which may interfere with or limit the proposed method.
- f. A description of QC procedures necessary to ensure sensitivity, accuracy and precision. This may include surrogate and QC spiking compounds, acceptance criteria, continuing laboratory proficiency testing, the use of SRMs, etc.
- g. Recommendations and conclusions. Item b through g above are critical if we must submit equivalency petitions to the Agency.
- h. I estimate of time/cost of conducting the method including special costs of reagents or standards required. The time estimates should include separate items for sample preparation, instrument calibration, software requirements, analysis and data reduction/assessment.

# Management

The studies will be managed as follows: The Vice President, Environmental Operations, will assign laboratory managers or project managers specific methods needing validation and approve the selection of the subsequent "team" members. The project manager or the laboratory manager will be designated for the preparation of the required reports.

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Page 8 of 8

#### REFERENCES

- Taylor, John K., Analytical Chemistry, 1983, 55, "Validation of Analytical Methods", 600A - 608A.
- 2. ACS Committee on Environmental Improvement, "Principles of Environmental Analysis", Analytical Chemistry, 1983, 55, 2210 2222.
- 3. Long, Gary L., Winefordner, J.D., "Limit of Detection: A Closer Look at the IUPAC Definition", Analytical Chemistry 1983, 55, 713A 724A.
- Kratochirl, Byron, Taylor John K., "Sampling for Chemical Analysis", Analytical Chemistry, 53, 1981, 925A - 938A.
- 5. Taylor, John K., "Quality Assurance of Chemical Measurements", Analytical Chemistry, 53, 1981, 1588A 1596A.
- 6. Glaser, John A., et-al., "Theory and Application of Method Detection Limit", Environmental Science and Technology, 1981, 51, 1426 1435.

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APPENDIX A

METHOD VALIDATION STUDIES

AR300622

Date: December 4, 1985

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### General Approach for the Validation of Analytical Methods by the Laboratory

#### Introduction

Historically our laboratory has determined the viability of published procedures by performing lab proficiency tests; i.e.-full analyte spikes are added to quadruplicate aliquots of laboratory pure water or "blank" soil, the samples are prepared/extracted and then analyzed by the appropriate instrumentation. The instrumentation would include GC/MS, GC, with appropriate detectors, and in the inorganic area, ICP/AAS/cold vapor AAS.

The laboratory proficiency testing program has been beneficial in demonstrating to ourselves and to interested clients that our applications of specified analytical methodologies are capable of producing acceptable data. The acceptable data is further characterized with statements of accuracy and precision; mean percent recoveries and standard deviations, respectively.

A necessary complement to the laboratory proficiency tests would be a formalized method detection limit study.

Before describing the rudiments of a recommended "Generic Method Validation Study," certain definitions of terms are required. John K. Taylor (1) of NBS presents the following definitions of the hierarchy of methodology; from the general to the specific:

- A Technique is a scientific principle, useful for providing compositional information.
- (2) A Method is a distinct adaptation of a technique for a selected measurement purpose.
- (3) A Procedure is composed of the written directions necessary to utilize a method.
- (4) A Protocol is the most specific name for a method and contains a set of definitive directions that must be followed, without exception, in order that the results be accepted for a given purpose.

Additionally, in an article entitled "Principles of Environmental Analysis" (2), a distinction is made between verification and validation:

- (1) Verification is a general process used to decide the capability of a method for producing accurate and reliable results.
- (2) Validation is an experimental process which involves external corresponding by other laboratories (internal or external) or methods or the list Lot 0 2 3 reference materials in order to evaluate the suitability of methodology.

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Our laboratory is embarking on a second generation of testing requirements which will serve to formally "validate" the "methods" we employ for new product offerings. The "Generic Method Validation Study" will serve to supply the data needed to satisfy ourselves and our clients that the laboratory's approach is sound. The impetus will be on the individual laboratories to prepare the specific experimental design, based on the method being validated. Additionally, the individuals actually performing the sample preparation, instrument calibration, analysis and data reduction processes will be required to utilize laboratory notebooks. The purpose of the laboratory notebooks is fourfold:

- (1) To record observations concerning problems encountered in applying the experimental design as written,
- (2) To note recommendations which may serve to eliminate the problems experienced,
- (3) To serve, with the experimental design, as a basis for the Standard Operating Procedures (SOPs) which will subsequently be required, and
- (4) To provide a basis for the preparation of an "Equivalency" petition to be submitted to the EPA.

(Note: as indicated above under the definitions of the hierarchy of methodology, a Procedure should not be able to be written until a Method has been utilized; i.e. - until the laboratory testing of the Method is accomplished and the details of the tests; problems/observations/recommendations, as written in laboratory notebooks, are evaluated).

In validating a method, the kinds of samples (matrices) to be processed should be clearly described. As a result of the validation process, statements of precision and accuracy will be generated. It should be realized that these data serve only as an estimate of the typical performance expected.

In being able to judge the suitability of a method, other factors have to be considered: sensitivity to interferences, limits of detection and useful range of measurement (1).

Interferences may come from two sources: those that are inherent in the matrix and laboratory artifacts, introduced during the sample processing.

By running appropriate method blanks and/or unspiked controls, the interferences can be characterized.

The concepts of detection limits and quantitation limits require elucidation (2).

- The Limit of Detection (LOD) is defined as the lowest concentration level that can be determined to be statistically different from a blank.

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- The Method Detection Limit (MDL) is the lowest concentration of analyte that a method can detect reliably in a sample or blank.

- The Instrument Detection Limit (ID) is defined as the smallest signal above background noise that an instrument is able to detect reliably.
- The Limit of Quantitation (LOQ) is defined as the level above which a specified degree of confidence may be otained for the quantitative results.

Our lab has historically used published detection limits or contract required detection limits. In situations where we are validating methods for which there are no detection limits (method or contractually defined), it will be our responsibility to correctly develop detection limits. It is important to understand the concepts since the reportable results will fall into different regions of reliability.

The "General Method Validation Study" write-up which follows is written for those methods which have already been written; the data produced from the study presents our application of the method. If methods are truly developed by us, the number of samples will be required to increase since statistical considerations suggest that at least six degrees of freedom (ordinarily seven measurements) should be involved at each decision point.

Classical validation processes involve the use of standard reference materials (SRM) after generating preliminary data. This approach is more viable when the SRM are similar in all respects to the test samples. The use of SRM may be appropriate as a final validation step if the number and type of analytes, and the matrix is the same. However, since many of the methods to be validated must encompass a variety of matrices and a cross-section of analytes, SRM may not be available. This will not preclude the use of those materials as part of a QA program to periodically insure us that our analytical systems are under control.

#### Generic Method Validation Procedure

The procedure being presented here is applicable for the GC/MS, GC, and inorganics laboratories. The purpose of the testing program is to generate precision, accuracy and recovery data on an aqueous and solid matrix, spiked with analytes of interest at one specified concentration. It should be used to gain experience and to demonstrate our laboratory's capabilities in applying procedures which have already been written; e.g. SW 846 Methods, Method 601, 602, etc. If our laboratory is truly developing a new method, another testing scheme would be applied.

Validation of an organic method using only water and sand matrices is judged to be suitable only for those instances where one or more surrogates can be used to monitor the effectiveness of the method in more complex matrices. For those organic methods where surrogates are not employed, testing additional matrices, e.g. - clay, planter's mix should be incorporated into the validation process 25

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A method validation study requires that laboratory notebooks be utilized in order to record any observations/problems encountered. Realistically, our SOPs should not be written until we have experience in applying the method being evaluated.

The conduct of, and the results from, each step are to be documented in laboratory notebooks. The notebooks should also serve to record any recommendations which can be made concerning a better application of the sample processing, analysis, or data evaluation steps. The steps to be taken in this validation process are as follows:

#### 1. Desk Top Review

The method as written is read by a chemist familiar with extraction/work up procedures and the instrumental detection systems required. During this reveiw, the chemist will particularly look for:

- A. Safety hazards.
- B. Applicability of available instrumental systems.
- C. New equipment/systems required that are not available.
- D. Discrepancies in the write up which do not appear to make sense from a chemical analysis standpoint. Exceptions to the write up need to be clearly identified.
- E. QA/QC requirements

#### 2. Preparation of Lab Plan

The lab plan is essentially, the testing approach to be taken and includes the proposed members of the "team" conducting the study and the specific exceptions, if any, to be taken from the method as written. The lab plan will be presented for approval to a review committee consisiting of Lee Myers, Chuck Bannerman, Ross Robeson and Bob Meierer.

#### 3. Preparation of Draft Method

The draft method will be written. Use of a xerographic copy from a standard manual is acceptable.

As an appendix to this draft method, the laboratory manager or project manager shall present the compounds to be spiked into the matrices to be tested. The analytes composing the spiking solutions should be all those (organic or inorganic) for which the method is being validated. AR300626

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Date: December 4, 1985

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Subsequent laboratory proficiency tests or standard reference materials, will be used on a routine basis to provide additional data on our application of the procedure.

#### 4. Laboratory Analysis

The matrices being evaluated are clean sand and laboratory pure water.

Method blanks consisting of aliquots of the sand and water are required.

Surrogate(s) are required for all organic procedures being evaluated.

When spiking these test samples, a minimum of one hour should elapse after spiking and thoroughly mixing and before the sample preparation process. Recommendations for modifications to volatile spiking requirements will be considered.

The spiked matrices shall be prepared and analyzed using the method write-up prepared under item 3 above. If our method differs from the published method, both must be run.

The spiking level to be analyzed in quadruplicate is:

An exact spiking level cannot be specified because the overall method recoverability is not known. Approximations of the recoverability can be made and used to prepare the spiking level. Alternatively, preliminary data points can be obtained by generating some recovery data on one or more spikes, using an estimate of a mid-level concentration.

#### 5. Detection Limit Run

After the data from section 4 (Laboratory Analysis above) is obtained, a formalized Method Detection Limit Study should be performed following the design specified by the EPA (for both water and sand matrices) in October 26, 1984 Federal Register.

#### 6. Summary Report Requirement

The written report, documenting the experimented effort, will be submitted to the Vice President, Laboratory Operations, for review. This report will include as a minimum:

a. Safety requirements for routine operation of the method in the laboratory.

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- b. A full description of the method including all procedures and equipment used. This description must highlight deviations from the method as written in the applicable government regulation or manual (e.g. SW-846 Manual, etc.).
- c. A description of the matrices tested.
- d. A comparison of results obtained with our method if different from the published method. Data should be tabulated to present actual results per test sample per compound/element and the mean recoveries and % RSD data. These reports should be as detailed as possible since they will serve a threefold purpose:
  - They will serve as the basis for the preparation of written SOPs,
  - They will be used in marketing efforts for new product offerings and will clearly demonstrate the extra effort which CompuChem takes in providing analytical data of the highest quality, and
  - Serve as the basis for documenting requests for equivalency of CompuChem methods to EPA published methods (if necessary).
- e. An assessment of any factors which may interfere with or limit the proposed method.
- f. A description of QC procedures necessary to ensure sensitivity, accuracy and precision. This may include surrogate and QC spiking compounds, acceptance criteria, continuing laboratory proficiency testing, the use of SRMs, etc.
- g. Recommendations and conclusions. Item b through g above are critical if we must submit equivalency petitions to the Agency.
- h. An estimate of time/cost of conducting the method including special costs of reagents or standards required. The time estimates should include separate items for sample preparation, instrument calibration, software requirements, analysis and data reduction/assessment.

#### Management

The studies will be managed as follows: The Vice President, Environmental Operations, will assign laboratory managers or project managers specific methods needing validation and approve the selection of the subsequent "team" members. The project manager or the laboratory manager will be designated to guide the effort and will be responsible for the preparation of the required reports.

AR300628

Date: December 4, 1985

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#### REFERENCES

- Taylor, John K., Analytical Chemistry, 1983, 55, "Validation of Analytical Methods", 600A - 608A.
- 2: ACS Committee on Environmental Improvement, "Principles of Environmental Analysis", Analytical Chemistry, 1983, 55, 2210 2222.
- 3. Long, Gary L., Winefordner, J.D., "Limit of Detection: A Closer Look at the IUPAC Definition", Analytical Chemistry 1983, 55, 713A 724A.
- Kratochirl, Byron, Taylor John K., "Sampling for Chemical Analysis", Analytical Chemistry, 53, 1981, 925A - 938A.
- 5. Taylor, John K., "Quality Assurance of Chemical Measurements", Analytical Chemistry, 53, 1981, 1588A 1596A.
- 6. Glaser, John A., et-al., "Theory and Application of Method Detection Limit", Environmental Science and Technology, 1981, 51, 1426 1435.

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#### APPENDIX B:

Resumes And Experience

Of Key Technical Personnel

# Robert E. Meierer Director Quality Assurance CompuChem® Corporation

Responsibilities:

Since 1983, Mr. Meierer has been the Director of Quality Assurance and responsible for assuring that all Corporate laboratories (CompuChem® Laboratories and ChemWest Laboratories) consistently produce high quality and reliable data and that all necessary certification and licensing requirements are met by the laboratories.

Education:

Mr. Meierer received an Associate degree in Industrial Chemistry from the Erie County Technical Institute in 1963, and an undergaduate degree in Chemistry from the State University of New York at Buffalo in 1971. He has taken advanced studies in Analytical Chemistry and Business Administration from the State University at Buffalo.

Experience: ....

Prior to joining CompuChem®, Mr. Meierer held positions as Laboratory Manager with Radian Corporation and as Department Head, Analytical Laboratory; Special Contamination Monitoring, The Carborundum Company from 1969 - 1980.

In his previous position with CompuChem® as Technical Development Scientist, Mr. Meierer was responsible for providing technical assistance to operational laboratories for procedure development and implementation and problem solving. Mr. Meierer has previously been employed with CompuChem® as Manager of Analytical Laboratories where he was responsible for directing the efforts for the Sample Preparation Laboratories, the Inorganic Laboratory, the GC Laboratory, and the Standards Laboratory.

Through the variety of laboratory positions Mr. Meierer had held, he has gained ten (10) years of experience in the interpretation of mass spectra gathered in GC/MS analysis. Additionally, Mr. Meierer has gained six (6) years experience in the preparation of extracts from environmental or hazardous

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Experience cont'd .:

waste samples. Further, he has gained three (3) years experience in organochlorine pesticide residue and PCB analysis, including clean-up procedures such as column chromatography, on environmental samples.

Publications:

Meierer, R.E., "Laboratory Data Credibility and Reliability," the paper presented in Milwaukee, Wisconsin on March 8, 1980, at the Federation of Environmental Technologists Conference.

Meierer, R.E., Myers R.L., Whitehead, R.J., "Quality Assurance Studies Based On Analytical Condition Codes," paper presented to the Fifth Annual EPA Contract Laboratory Program Conference, U.S. EPA, August 1, 1985.

Meierer, R.E., "GC/MS: Applications For The Determination of Organic Constituents In Hazardous Waste," paper presented at the Twelfth Annual Conference on Waste Technology, NSWMA, October 18, 1983.

Meierer, R.E., Ragsdale P.L., and Mills, P.E., "Quality Assurance of Support Functions In A Large Hazardous Wastes Analytical Laboratory," paper presented before the division of Environmental Chemistry, American Chemical Society, March 29, 1982.

Shaffer, P.T.B., Meierer, R.E., McGee, C.D., "Virus Recovery From Natural Water" <u>JAWWA</u>., 69 (10), 528-531 (1977).

Cook, G.A., Meierer, R.E., and Shields, B.M. "Combustibility Tests on Several Flame-Resistant Fabrics in Compressed Air, Oxygen Enriched Air, and Pure Oxygen." Textile Research, 37:591 (1967).

Cook, G.A., Meierer, R. E., Shields, BM., and Nevins, H.E. "Effects of Gas Composition on Burning Rates Inside Decompression Chambers at Pressures Up To 350 Feet of Sea Water." Paper presented at 54th Annual Meeting, Under-Ocean Technology (Labelle 372 1967 (Published by the Compressed Gas Association).

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#### Robert J. Whitehead Manager of Quality Assurance CompuChem® Laboratories

Responsibility:

As the Manager of Quality Assurance, Mr. Whitehead is responsible for managing the Environment QA and Forensic Drug Testing QA operations.

Education:

Mr. Whitehead received an undergraduate degree in Biology, with a secondary emphasis in Chemistry, from the University of North Carolina at Chapel Hill. Mr. Whitehead has also participated in a number of Continuing Education Programs and symposia associated with Statistical Quality Control, Analytical QA/QC, Analytical Techniques, Waste Testing and Quality Assurance, Quality Circles Concepts, and Advanced Leadership Training.

Experience: \_\_\_ .

Before his promotion to Manager of Quality Assurance, Mr. Whitehead was employed at CompuChem® Laboratories as a Sr. QA Specialist, responsible for ensuring that data generated by all lab stations complied with established acceptance criteria. Prior to this, Mr. Whitehead was employed at CompuChem® Laboratories as a GC/MS Operator, with responsibility for the operation of a GC/MS system, spectral interpretation, and quantitative data analyses. Prior to joining CompuChem® Laboratories on a full time basis, Mr. Whitehead had been employed in the GC/MS Lab on a part-time basis, during his senior year in college.

Mr. Whitehead has 2 years of experience in the operation of the GC/MS/DS on environmental samples and 8 years of experience in the interpretation of mass spectra gathered in GC/MS analysis. Mr. Whitehead also has 2 years of experience using the purge and trap

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Experience cont'd.:

technique for volatile organics and 1 year of experience in the preparation of extracts from environmental or hazardous waste samples. Additionally Mr. Whitehead has 5 years experience in conducting QA systems and performance audits, and has been directly involved in the development of numerous QA Project Plans and QA Program Plans following QAMS-005/80 and QAMS-004/80 guidelines.

Publications:

Whitehead, R. J., "Laboratory Data Credibility and Reliability," the paper presented in Milqaukee, Wisconsin on March 8, 1980, at the Federation of Environmental Technoligists Conference.

Whitehead, R. J., "Statistical Quality Control for the Analytical Laboratory," <u>Proceedings</u> from the <u>Analytical Techniques and Residuals Management in Water Pollution Control Specialty Conference</u>, Water Pollution Control Federation, April 20, 1988.

#### Manhar R. Amin Senior Standards Technician CompuChem® Laboratories

Responsibility:

As Senior Standards Technician, Mr. Amin is responsible for the operation of the standards function to provide the various laboratories (GC, GC/MS, Inorganics) with timely and accurately prepared standards.

Education:

Mr. Amin received an undergraduate degree in Microbiology with a minor in Chemistry from S.B. Garda College, Navsari, India in 1963.

Experience:

From 1963 - 1979, Mr. Amin was employed as
Junior Scientific Officer of Quality Control
Laboratory with Alembic Chemical Works in
India. From 1979 - 1981, Mr. Amin was
employed as a Chemistry Laboratory Assistant
with Wyeth Laboratories.

Mr. Amin joined CompuChem® Laboratories in February 1982' as a Senior Laboratory Assistant. Since then he has held the position of Standards Technician, then later he was promoted to his current position in June 1986'.

# Joe Bumgarner Manager of Sample Preparation Laboratory CompuChem® Laboratories

Responsibilities:

In 1988 Mr. Bumgarner was promoted to his present position where he manages the preparation of Samples in his department, as well as managing the Organic Characterization Laboratory where the analyses of Total Petroleum Hydrocarbons (TPH), Total Organic Carbon (TOC), and Total Organic Holides (TOX) is performed.

Education:

Mr. Bumgarner received an undergraduate degree in Biology from Garner-Webb College in 1985.

Experience:

Mr. Bumgarner joined CompuChem® in May, 1985
as Senior Laboratory Assistant. In October
1985, he was promoted to Supervisor of the
Sample Preparation Laboratory, where he was
responsible for the supervision of the
activities of the Sample Preparation Lab
ensuring that high quality work was performed
in a timely and efficient manner.

Debra L. Stanley
Supervisor Sample Preparation Laboratory (2nd shift)
CompuChem® Laboratories

Responsibility:

Ms. Stanley was promoted to her present position on June 1, 1986 and is responsible for the supervision of the activities of the Samples Preparation Laboratory ensuring that high quality work is performed in a timely and efficient manner.

Education:

Ms. Stanley received an A.A.S. degree in Medical Technology from Career Academy, Atlanta, GA in 1972.

Experience:

From 1972 to 1979, Ms. Stanley was employed as Medical Technician at Spring Hope Clinic. From 1976 to 1979, she was employed as Pediatric Nurse with Drs'. Poole, Winslow, and Brown.

Since joining CompuChem® on May 12, 1980, Ms. Stanley has held positions as Laboratory Technician, GC/MS Operator Trainee, and GC/MS Operator.

# Candace Jacobs Technician IV - Environmental CompuChem® Laboratories

Responsibility:

Currently, Ms. Jacobs is responsible for the extract of environmental samples of various matrices.

Education:

Ms. Jacobs completed her Junior year at the University of Texas at Austin, one year at North Carolina State University and she is lacking 80 semester hours towards her B.S. degree in biochemistry.

Experience:

Before joining CompuChem® Ms. Jacobs did college chemistry laboratory work which is related to her current position.

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#### Bernard Dickens Technician III CompuChem® Laboratories

Responsibility:

Currently Mr. Dickens is a Technician III, responsible for the extract of environmental

samples of various matrices.

Education:

Mr. Dickens received 2 years of college as a Biology major at Saint Augustine's College.

Experience:

Mr. Dickens has two years of Planning, Production and Control experience prior to his

current position.

Linda A. Pittman
Technician IV
CompuChem® Laboratories

Responsibilities:

Ms. Pittman is a Technician IV, responsible for the extraction of environmental samples of varied matrices using liquid-liquid and solid-liquid techniques. She also assists in training other members of the laboratory staff.

Education:

Ms. Pittman received a high school equivalency diploma in 1967.

Experience:

Prior to joining CompuChem®, Ms. Pittman was employed with Burlington Industries as a Machine Operator in 1976.

Ms. Pittman joined CompuChem® in 1980 and was employed as Senior Lab Assistant and Glassware Preparer in the Sample Preparation Laboratory. Ms. Pittman has three (3) years of experience in the preparation of extracts from environmental or hazardous waste samples.

Appendix B Revision No. 4

Date: October 17, 1988

Eddie Howard Thompson Technician III CompuChem® Laboratories

Responsibility:

Mr. Thompson's responsibility as a Technician

III is to extract samples.

Education:

Mr. Thompson received his high school diploma

at Valley High School, in Sacramento CA,

1983.

Experience:

Before being promoted to his current position, Mr. Thompson spent two (2) years in Glassware Preparation and one (1) year

extracting samples.

Appendix B. Revision No. 4

Date: October 17, 1988

Zelphia Lipscomb Technician IV CompuChem® Laboratories

Responsibility:

Ms. Lipscomb is a Technician IV, responsible for the extraction of environmental samples of

varied matrices using liquid-liquid and

solid-liquid techniques.

Education:

Ms. Lipscomb received her high school diploma in 1974 and was pursuing accounting courses at Durham Technical College in 1975 - 1977.

Experience:

Prior to joining CompuChem®, Ms. Lipscomb was employed by Peoples Life Insurance Company as

a CRT Operator during the year of 1982.

#### Mark Riggs Technician III CompuChem® Laboratories

Responsibility:

Mr. Riggs responsibility as a Technician III is to extract samples.

Education:

Mr. Riggs received his B.A. Degree in Foreign Languages in 1975 at the University of North Carolina, Asheville, and received his M.A. in Foreign Languages in 1977 from UT Knoxville.

Experience:

Prior to joining CompuChem®, Mr. Riggs was employed by Northern Telecom as Quality Control Inspector from July, 1985 to December, 1985.

Mr. Riggs joined CompuChem® in January 1986'. Before being promoted to his current position, he was a Senior Laboratory Assistant responsible for the preparation of samples with different matrices for analysis by GC and GC/MS.

#### Melody L. Enscore Technician IV CompuChem® Laboratories

Responsibilities:

As a Senior Environmental Sample Prep.
Technician, Ms. Enscore is responsible for laboratory procedures involving chemical extractions of various matrices and column chromatography (clean-up) and other activities to prepare soil & water samples for gas chromatography analysis. She is also responsible for laboratory inventory maintenance and training lower level technicians. Ms. Enscore develops problem-solving strategies for problematic samples.

Education:

Ms. Enscore has a B.A. in English/Comp. Literature (UNC-Chapel Hill, 1985), and a M.A. in Comparative Literature (UNC-CH, 1988).

Experience:

Ms. Enscore worked as a lower level technician, at CompuChem® before being promoted to current position.

Ms. Enscore took courses in High School and College Chemistry coursework that is related to current position.

#### Carrie Beth Robertson Technician III CompuChem® Laboratories

Responsibilities:

Currently, Ms. Robertson is responsible for performing wet chemistry techniques, including liquid-liquid and liquid-solid extractions. She also performs column chromatography procedures.

Education:

Ms. Robertson was in the Laboratory Technician Program from TCA for one (1) year. She is presently attending Elon College to obtain a B.S. in Chemistry.

Experience:

Before joining CompuChem® Ms. Robertson worked at Roche Biomedical approximately three years in the RIA Department. She was responsible for running the tests: T3 up-, T4 RIA, Dioxin and B12-Folate. Ms. Robertson made judgemental calls from QC levels.

# Cynthia Bowden Technician II CompuChem® Laboratories

Responsibilities:

As a Technician II, Ms. Bowden is responsible for using lab techniques, procedures, wet chemistry techniques, chromatography procedures, and she prepares the related paperwork.

Education:

Ms. Bowden received a B.S. degree in biology at NCCU in 1984. She has two years into her Master's degree - biology at North Carolina Central University.

Experience:

Before being promoted to her current position, Ms. Bowden was a Sample Prep Technician Trainee. She was responsible for developing an understanding of lab techniques, procedures, and to learn all quality control batches to be prepared with associated samples and to recognize differences and prepare related paperwork.

Ms. Bowden had taken relative lab courses in curriculum for major - minor in Chemistry.

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Date: October 17, 1988

Rebecca L. Howell Technician III CompuChem® Laboratories

Responsibility:

Currently Ms. Howell is responsible for the extraction of herbs/pest./semi-vol. from soil/water media in the preparation for analysis by GC's/MS's or GC.

Education:

Ms. Howell graduated from Heidelberg College in May 87' with a B.S. in Environmental

Biology/English.

Experience:

Before joining CompuChem® Ms. Howell did lab work in a classroom atmosphere, and she was a Lab Prep Technician from August 1987 to December 1987, at Heidelberg college which is

related to her current position.

#### Anthony D. Rice Technician IV CompuChem® Laboratories

Responsibility:

Currently Mr. Rice is responssible for the extract of environmental samples of various matrices.

Education:

Mr. Rice is a high school graduate of 1980 and has pursued studies in Accounting at Durham Technical Institute 1981 - 1982.

Experience:

Mr. Rice worked with various temporary employment agencies from January of 1985 - June of 1985. He joined CompuChem® July of 1985 as a Glassware Preparer where he was responsible for the preparation and inventory of glassware for the laboratories.

Anh T. Chan Manager Data Review CompuChem® Laboratories

Responsibilities:

Ms. Chan has held the position of Manager of Data Review since January 1987. In this capacity she is responsible for ensuring the steady flow of the reviewed data from the GC/MS lab to the Production, Planning and Control Departments in order to meet the projected deadline, to make technical judgements and decisions on anomalous data, to maintain close contacts with the Quality Control and Quality Assurance Departments with a view to producing the highest quality data, to serve as a feedback mechanism to the GC/MS lab, and ensure completion of data without delay.

Education:

Ms. Chan received her B.A. degree in General Science with emphasis in Chemistry from Brandeis University in 1977.

Experience:

Prior to joining CompuChem®, Ms. Chan was employed by the Research and Analytical Laboratory, School of Public Health, University of North Carolina, Chapel Hill as a Senior Research Technician from July, 1979 - October, 1979.

Ms. Chan joined CompuChem® November 1979 as a GC/MS Operator and also held the position of Senior GC/MS Operator and Spectroscopist before being promoted to Assistant Manager of GC/MS. In October 1986 Ms. Chan was responsible for the supervision of Environmental GC/MS data review. She was then promoted to Manager of Data Reveiw.

- Angela Childress
Manager, Production Planning & Control
CompuChem® Laboratories

Responsibility:

As Manager, of the Production Planning and Control Department, Ms. Childress is responsible for managing the daily activities of the production and scheduling function to ensure schedules or commitments are met.

Education:

Ms. Childress received a Master of Business Administration degree at the University of Arkansas, July 1983. She received a BS degree in Industrial Management from the University of Arkansas, May 1980.

Experience:

From August 1987 to November 1988 Ms. Childress was employed as an Industrial Engineer at CompuChem® Laboratories where she initiated the first cost analysis for 80% of the Environmental product line. She also served as management trainer for the Zenger-Miller Supervisory Training Program. She initiated the first labor standards for use in scheduling, capacity planning, and lab floor control, and she coordinated with the Production Planning & Control manager in establishing the first centralized scheduling program. Ms. Childress developed work station lay-outs within the environmental and clinical laboratories to increase employee efficiency through improved flow. Ms. Childress coordinated with and assisted the Coopers & Lybrand consulting team in analysis of the current environmental laboratory operation.

From July 1987 to August 1987, Ms. Childress was a self employed Management Consultant at Johnson & Johnson - Chicopee Division, Benson, NC, where she conducted a warehouse utilization study that reviewed space allocation, personnel/equipment utilization, and product flow with recommendations from increased efficiency, improved labor utilization, and smoother material flow.

From April 1984 to June 1987 Ms. Childress worked as an Industrial Engineering Supervisor at Johnson & Johnson - Chicopee Division, North Little Rock, AR. At this company she monitored N.L.R. incentive plan affecting 110 wage personnel. She supervised a technician and an incentive clerk, and determined labor and production rates for new and revised product company.

Experience cont'd.:

Ms. Childress also performed I.E. project work for 2 plants in N.L.R. (400 people), 1 plant in Camden, AR (150 people), & 1 plant in Benson, NC (200 people). She also served as speaker for local schools.

From July 1980 to February 1984 Ms. Childress was employed as a Work Management Coordinator, at Little Rock Municipal Water Works in Little Rock, AR. She designed and implemented a computer generated work order system used by 60 field personnel. Ms. Childress developed and conducted training for a new work order system, standards, and Quality Circles. She designed & implemented a "real time" inventory control system and served as the first Quality Circles facilitator at the facility. Ms. Childress established initial 49 times standards & optimum work methods for field personnel and served as speaker for the National Water Works Association conference in Las Vegas, the Central Arkansas Compensation Association, and the Arkansas Water Managers' Association.

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#### Ann Marie Flaherty Manager, Report Preparation/Tech Review CompuChem® Laboratories

Responsibilities:

On November 14, 1988 Ms. Flaherty became Manager of the Report Preparation/Technical Review Department, responsible for the integration, technical review and audit, word processing and full service deliverables package of the data generated by Compuchem's analysis procedures.

Education:

Ms. Flaherty received an undergraduate degree in Industrial Relations/Psychology from the University of North Carolina at Chapel Hill in 1982.

Experience: . ..

Ms. Flaherty was employed at IBM in 1982 as a PP&C Clerk. Prior to being promoted to her current position, Ms. Flaherty held positions as Scheduling Clerk, Report Integration Clerk and Supervisor Scheduling and Sample Saver. Ms. Flaherty was promoted to Manager of Production Planning and Control on October 20, 1986 and was responsible for managing daily activities of the Production and Scheduling functions.

She has attended several seminars including Fundamentals of Supervision (24 hours) at Capital Associated Industries in 1985, Advanced Leadership Development Program (27 hours) at CAI in 1985, and Zenger Miller Frontline Leadership Training (24 hours) in 1988.

#### Susan Bass Manager Volatile Laboratory CompuChem® Laboratories

Responsibilities:

As Manager of the Volatile Laboratory, Ms. Bass is responsible for the preparation and analysis of environmental volatile samples utilizing GC/MS and for the generation of complete data packages. She is responsible for for managing the multi-shift Volatile taboratory ensuring that timely and accurate production is achieved.

Education:

Ms. Bass received her B.S. degree in Chemistry from Meredith College in 1978.

Experience:

Prior to working for CompuChem®, Ms. Bass was employed by the North Carolina Department of Agriculture as a Chemical Analyst from 1979 - 1980. Also, she was employed by Becton Dickerson and Company as a Research Assistant from 1980 - 1981.

Ms. Bass joined CompuChem® April of 1981 as a Junior GC/MS Operator and held positions of GC/MS Operator and Senior GC/MS Operator before being promoted to her current position of Project Volatile Manage.

Bruce H. Rohrbach
Manager Inorganics Laboratory
CompuChem® Laboratories

Responsibility:

Mr. Rohrbach joined CompuChem® on February 2, 1987 and is responsible for managing the Inorganics Laboratory ensuring the production of accurate data in a cost and time effective manner so that laboratory goals are met.

Education:

Mr. Rohrbach received an BA degree in Chemistry from West Chester State University in 1972.

Experience:

From 1986-1987 Mr. Rohrbach was Inorganics Laboratory Manager with Ecology and Environment, Inc. Additionally, he was employed as a Research Chemist with Allied Corporation from 1980-1986. Mr. Rohrbach was employed with Allentown Testing Lab as Chief Chemist/Laboratory Supervisor (1975-1980); Chemical Testing Laboratory Manager (1973-1975); and Analytical Chemist (1972-1973).

William R. Desjardins Manager, GC Projects CompuChem® Laboratories

Responsibiliies:

Mr. DesJardins is employed at CompuChem® as Manager of GC Projects, in the GC Laboratory, with responsibility for the development and application of GC methods for samples requiring analysis using ECD, FID, NPD and PFD detectors.

Education:

Mr. DesJardins received a B.S. degree in Biology from Guilford College in Greensboro, NC in 1980.

Experience:

Prior to coming to work at CompuChem®, Mr. DesJardins was employed by the Occupational Health Studies Group as a Lab Technician, where his duties included performing GC analysis of dust, solvent and air samples.

Mr. DesJardins has I year of experience in the preparation of extracts from environmental or hazardous waste samples and 5 years experience in organochlorine pesticide residue and PCB analysis, including clean-up procedures such as column chromatography on environmental samples.

Charles T. Mann
Supervisor of the GC/MS Lab
CompuChem® Laboratories

Responsibilities:

On 2/29/88 Mr. Mann became Supervisor of the GC/MS Laboratory, where he is responsible for ensuring that the production of the Semi-Volatile Laboratory on a single shift is conducted in a timely and accurate manner. This includes coordinating the production effort with the Supervisors on other shifts. Mr. Mann is responsible for evaluating and developing methods for improving the quality and quantity of the data produced. Other responsibilities include: providing technical guidance and input for new contract requirements and/or new products; planning and scheduling work assignments according to analysis requirements; assigning individual work schedules based on analysis requirements and capabilities of the department staff; being responsible for interviewing, selecting orienting, and training new employees; determining training needs of current employees and defining a plan of action to address the training requirements; providing recommendations for promotions and lateral transfers; conducting performance appraisals, recommending merit increases and reviewing merit increases with employees; being responsible for communicating and ensuring that all departmental employees understand and adhere to all company policies and procedures; maintaining an awareness of all Federal, State, and local rules and regulations that pertain to employment practices, i.e., Wage and Hour laws, Equal Employment Opportunity, and OSHA regulations; and being responsible for safety attitudes and practices; and for the overall houskeeping of the Semi-Volatile Laboratory.

Education:

Mr. Mann received a B.A. in Chemistry from Wake Forest University in 1985.

Experience:

Mr. Mann joined CompuChem® as a GC/MS
Technician during the summer of 1984 and on
weekends prior to his permanend Amgleynes 5 A

Experience cont'd.:

May 28, 1985. He has over one (1) year of experience in the operation of a GC/MS/DS on environmental samples. Mr. Mann was promoted to this present position on June 3, 1986 where he was responsible for performing timely and accurate analysis of samples using GC/MS.

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Roy M. Sutton, Ph.D. Developmental Chemist CompuChem® Laboratories

Responsibilities:

Dr. Sutton is responsible for the accurate and complete technical review of the data generated by the GC/MS Laboratory in order to assure the highest quality data, applying stated quality control policies and maintaining the records necessary to support these policies.

Education:

Dr. Sutton received a BS degree in Entomology from Clemson University in 1974 and in 1978 he received a Ph.D. in Entomology from Clemson University.

Experience:

Previously Dr. Sutton was responsible for the accurate and complete technical review of the data generated by the GC/MS laboratory in order to assure the highest quality data, applying stated quality control policies and maintaining the records necessary to support these policies. Also he is responsible for final EPA case audits.

Prior to joining CompuChem®, Dr. Sutton was employed with Burlington Industries, Inc., Corporate Research and Development, where he gained experience in infrared spectrophotometry, gas chromatography, mass spectrometry, and nuclear magnetic resonance.

Through the various laboratory positions Dr. Sutton held, he has gained 6 years of experience in the interpretation of mass spectra gathered in GC/MS analysis. Additionally, he has 4 years of experience in the operation of the GC/MS/DS on environmental samples and 2 years of experience in the preparation of extracts from environmental or hazardous waste samples. Dr. Sutton also has 3 years of experience using the purge and trap technique for volatile organics.

L. Richard Flynn
Developmental Chemist
CompuChem® Laboratories

Responsibilities:

Mr. Flynn is responsible for the accurate and complete technical review of the data generated by the GC/MS Laboratory in order to assure the highest quality data, applying stated quality control policies and maintaining the records necessary to support these policies.

Education:

Mr. Flynn has an undergraduate degree in Chemistry from the University of North Carolina at Chapel Hill.

Experience:

Prior to coming to CompuChem®, Mr. Flynn was employed at the Research Triangle Institute where his duties included performing general analytical laboratory functions involved with trace organic analysis.

Through the various laboratory positions Mr. Flynn held, he has gained 4 years of experience in the operation of the GC/MS/DS on environmental samples. Additionally, he has 2 years of experience in the interpretation of mass spectra gathered in GC/MS analysis. Also, he has 6 months of experience using the purge and trap technique for volatile organics.

## James T. Chambers Manager, Laboratory Instrumentation CompuChem® Laboratories

Responsibilities:

Mr. Chambers is the Manager of Laboratory Instrumentation and is responsible for planning, directing, and coordinating the operations of the Laboratory Instrumentation Department.

Education:

Mr. Chambers received his Bachelor of Science degree in Business Administration from Troy State University in Montgomery, Alabama. He received extensive training in electronics at the USAF Technical School, at Keesler AFB.

Experience:

Prior to joining CompuChem® Mr. Chambers was employed as a Systems Engineer with the Finnigan Corporation for 5 years. His responsibilities required specialized skills in electronic circuitry, the application of laboratory instrumentation, and performance reviews.

## Diana Scammell Technical Marketing Project Manager CompuChem® Laboratories

Responsibilities:

Ms. Scammell is responsible for providing guidance on special projects by reviewing client requirements in conjunction with the laboratory capacity and subsequent management of the project. This includes: review of request for proposals (RFPs), coordinating scope of work with the laboratory, designing reporting format, and resolving associated inquiries.

Acts as a technical liaison between CompuChem® Laboratories and the client, investigate and resolve technical inquiries. Also acts as technical liaison between the Marketing Department and the laboratories.

Education:

Ms. Scammell attended Virginia Polytechnic Institute & State University in Blacksburg, VA. 96 hours towards Biology/Animal Science.

Experience:

Prior to joining CompuChem® Laboratories, Ms. Scammell was a Laboratory Technician for Virginia Polytechnic Institute & State University where she collected water & sediment samples and performed routine chemical analysis.

Before being promoted to Technical Marketing Project Manager, Ms. Scammell held the position of Environmental Projects Manager and Technical Review Specialist.

### Michael Mattocks Data Review Specialist CompuChem® Laboratories

Responsibilities:

As a Data Review Specialist Mr. Mattocks is responsible for assuring the technical quality of commercial data by performing technical audits and monitoring laboratory trends.

Education:

Mr. Mattocks received a B.S. in Chemistry from North Carolina Central University in 1986.

Experience: - ...- - ... From February 1983 to September 1986, Mr. Mattocks was employed as Lab Technician with NIEHS. From September 1986 to May 1986, Mr. Mattocks was employed with Duke University as Laboratory Assistant.

> Mr. Mattocks joined CompuChem® as GC/MS Trainee on June 29, 1986. On March 30, 1987, he was promoted to GC/MS Operator and was responsible for analyzing and interpreting samples using GC/MS.

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\_\_\_ E. Robin Nowell
Data Review Specialist
CompuChem® Laboratories

Responsibilities:

As a Data Review Specialist, Ms. Nowell the assures technical quality of commercial data by performing technical audits and monitoring laboratory trends.

Education:

Ms. Nowell attended NC State University from 1974 to 1981, working toward a B.S. Degree in Zoology and Chemistry.

Experience:

Prior to joining CompuChem®, Ms. Nowell worked as a Laboratory Technician with Burroughs: Wellcome from November, 1981 - April, 1982. Prior to her promotion to Sr. Technical Reviewer, Ms. Nowell has held various positions at CompuChem®. Ms. Nowell was hired as a Biomedical Technician and was soon promoted to GC/MS Operator Trainee and was later promoted to the positions of GC/MS Operator, then to Technical Reviewer.

Ms. Nowell has 21/2 years experience in the operation of GC/MS/DS on environmental samples and 6 months experience in the screening and extraction of biomedical samples.

Stephanie D. Wagner Data Review Specialist CompuChem® Laboratories

Responsibilities:

As a Data Review Specialist, Ms. Wagner is responsible for specializing in volatile sample review, but trained in all analyses. She assess mass spectra for compound list hits and unknowns. Ms. Wagner makes final decisions on reinjection and repreparation of samples. She also writes laboratory notices to explain irregularities to clients. Ms. Wagner is involved in sample scheduling and tracking and interacts with operators and QA department to ensure completeness and quality of data. She also audits data reviewed by operators, and writes standard operating procedures.

Education:

Ms. Wagner received a B.S. Degree in Chemistry from North Carolina State University in May 1984 with a Computer Science Minor.

Experience:

From October 1985 to July 1987 Ms. Wagner worked as a GC/MS Operator Trainee. In June 1986 she was promoted to GC/MS Operator, where she analyzed semivolatile EPA samples as well as commercial BNA samples using a Finnegan OWA. Ms. Wagner mastered manual tuning, basic instrument repair and troubleshooting. She worked in Forensic Drug Testing GC/MS Laboratory for three (3) months, performed first level data review and helped train new personnel.

From July 1984 to September 1985 Ms. Wagner was employed at Research Triangle Institute as an Organic Chemist I, where she synthesized and analyzed organic compounds, mainly for the National Institute on Drug Abuse contract. She ensured the quality of final product using NMR, IR, UV, and optical rotation. Ms. Wagner also utilized general bench chemistry techniques plus HPLC work with peptides.

Elsie S. Byrd Sr. Data Review Specialist CompuChem® Laboratories

Responsibilities:

Ms. Byrd is responsible for performing full review of GC/MS data and laboratory deliverables for accuracy and completeness.

Education:

Ms. Byrd has a degree in Chemical Engineering from the Mapua Institute of Technology, Manila, the Philippines.

Experience:

Before her promotion Ms. Byrd held positions as Sr. GC/MS Operator, GC/MS Operator, GC/MS Operator Trainee, and Sr. Laboratory Assistant. Prior to joining CompuChem®, she was employed at Hercules, Inc. as a Sales Service Engineer, Research & Development Engineer and, Production Engineer.

Through the variety of positions Ms. Byrd has held, she has gained 3 years and 15 months experience with CompuChem® & remaining previous employer in the preparation of extracts. She has 3 years of experience in the operation of the GC/MS/DS on environmental samples.

## Linda L. Fowler Senior Data Review Specialist CompuChem® Laboratories

Responsbilities: ...

Ms. Fowler's primary responsibility is to perform full review of GC/MS data and laboratory deliverables for accuracy and completeness. She ensures that all data complies with contractural/customer requirements and internal standard operating procedures.

Education:

Ms. Fowler received a B.S. in Medical Technology from the University of Oklahoma, in conjunction with the Texas Medical Center, Houston, Texas.

Experience:

Prior to employment at CompuChem®, Ms. Fowler was employed as GC/MS Operator at Oklahoma Children's Memorial Hospital. Ms. Fowler has over ten (10) years work experience in a laboratory environment.

Additionally, Ms. Fowler has done GC/MS research and development, at Baylor College of Medicine, in Houston, TX.

### Sarah A. Hubbard Senior Data Review Specialist CompuChem® Laboratories

Responsibility:

Ms. Hubbard is responsible for performing full review of GC/MS data and laboratory deliverables for accuracy and completeness.

Education:

Ms. Hubbard has a B.E. degree in Chemical Engineering from Vanderbilt University and an M.S. in Analytical Chemistry from the University of New York in Binghamton.

Experience:

Prior to her promotion, Ms. Hubbard was a Sr. GC/MS Operator in the GC/MS Laboratory. Prior to joining CompuChem®, Ms. Hubbard was employed as a scientist with Northrop Services, Inc., where she employed GC in the analysis of air pollutants. She has also been employed with I.B.M. as a Chemist and as an Associate Engineer.

Through the various laboratory positions she held, she has gained 2½ years of experience in the operation of the GC/MS/DS on environmental samples. Additionally, she has one year of experience using the purge and trap technique for volatile organics.

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Date: October 17, 1988

Stephanie D. Wagner Data Review Specialist CompuChem® Laboratories

Responsibilities:

As a Data Review Specialist, Ms. Wagner is responsible for assuring the technical quality of commercial data by performing technical audits and monitoring laboratory trends.

Education:

Ms. Wagner received a B.S. Degree in Chemistry from North Carolina State Unviersity in 1984.

Experience:

From July 1984 to September 1985 she was employed as Organic Chemist at Research Triangle Institute.

Ms. Wagner joined CompuChem® on October 14, 1985 as GC/MS Trainee. She was promoted to GC/MS Operator June 9, 1986 and is responsible for analyzing and interpreting samples using GC/MS.

Jeanne C. Alston Final Technical Reviewer CompuChem® Laboratories

Responsibilities:

On December 14, 1988 Ms. Alston joined CompuChem® Laboratories as a Final Technical Reviewer, responsible for the review of EPA and commercial organic samples and commercial. Commercial CLP and EPA inorganic samples such that adherence to contract protocols and internal quality guidelines are met. She resolves issues/incidents noted in the review process with the director/manager to ensure internal quality of deliverable data. Ms. Alston also documents and tracts the issues/incidents noted in the review process to communicate with lab managers. Ms. Alston is also responsible for interpretation of current contract requirements and current statement of work documents.

Education:

Ms. Alston received a B.S. degree in chemistry from the University of North Carolina in Chapel Hill, NC on August 12, 1985.

Experience:

Prior to joining CompuChem®, Ms. Alston was employed by Triangle Laboratories being responsible for the extraction and clean-up (via liquid chromatography techniques) of dioxin/furan samples. Later she learned to operate a VG-, magnetic high resolution GC/MS for dioxin/furan analysis, then she learned to operate a VG-low resolution quadropole GC/MS for volatile analysis. Ms. Alston trained again in the wet lab to learn SOPs for the extraction of SV and Pesticide samples, afterwhich, she rotated between positions as needed. Ms. Alston's other responsibilities included the preparation and spiking of XAD traps and VOST (volatile organic sampling train) tubes.

### Janet C. Garrett Technical Reviewer CompuChem® Laboratories

Responsibilities:

In October 1984, Ms. Garrett was promoted to current position of Technical Reviewer which involves final review of environmental analytical data packages for adherence to contract protocols, laboratory operating procedures, and quality control guidelines. Ms. Garrett specializes in EPA/platinum organic product line, with over four years of data review experience.

Education:

Ms. Garrett received a B.S. degree in biology, minor in chemistry from Appalachian State University, Boone, North Carolina in 1981.

Experience:

Ms. Garrett began employment at CompuChem® in February 1983 as a biomedical technician in the clinical division and was responsible for extraction, screening, and GC/MS analysis of biological fluids for detection of drugs of abuse.

In October 1983, Ms. Garrett was promoted to volatile GC/MS Operator in the environmental division. She analyzed water and soil samples on Finnigan OWA GC/MS instruments using purge and trap method and performed initial data review including spectral interpretation.

Prior to joining CompuChem®, Ms. Garrett was employed for 1 1/2 years as a medical technologist at Roche Biomedical Laboratories, Inc., Burlington, NC, where she performed clinical diagnostic testing on biological fluids using radioimmunoassays.

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Date: October 17, 1988

Betty J. Andershock Final Technical Reviewer CompuChem® Laboratories

Responsibility:

On 7/11/88 Ms. Andershock became a Technical Reviewer, in the Technical Review Department. Ms. Andershock is responsible for the review of all environmental lab data to ensure its quality and contract compliance. She resolves issues noted in the review process and communicates with lab managers. Ms. Andershock is also responsible for the interpretation of current contract requirements and current SOW documents. Ms. Andershock writes narratives with each case explaining data.

Education:

Ms. Andershock received a B.S. degree in Zoology with minors in Chemistry and Psychology from Marshall University in 1986.

Experience:

From 9/14/87 - 07/08/88 Ms. Andershock worked as a GC/MS Operator Semivolatile - CompuChem®. She was responsible for the analysis of pesticides, base neutrals, acids, and semi 2's. Ms. Andershock was also responsible for the interpretation of data to ensure CLP and internal laboratory quality guidelines. She has the ability to perform analysis using Finnigan OWA.

From 7/14/87 - 9/14/87 Ms. Andershock worked as a Sr. Inorganic Technician at CompuChem® Labs. She was responsible for performing all inorganic prep procedures. Ms. Andershock maintained complete control of cyanide and phenol distillation and analysis. She was responsible for analyzing cyanide and phenol by use of technicon, mercury on video 12 using cold vapor technique, along with some training on video 22 AA. Ms. Andershock was responsible for the interpretation of all data and ensuring contract complicance in the review process.

John P. McConney Final Technical Reviewer CompuChem® Laboratories

Responsibilities:

On 7/21/86 Mr. McConney became a Final Technical Reviewer. In the Technical Review Department he is responsible for the final technical review of EPA samples, ensuring adherence to contract protocols and that internal quality guidelines are met. Mr. McConney is also responsible for knowing current contract requirements and statements of work, as well as lab methodologies, SOPs, and deliverable requirements. He is responsible for data review which includes evaluation of raw sample data, raw QC data, standards, and supporting data. Mr. McConney is also responsible for the production of the Case Narrative.

Education:

Mr. McConney's educational background consists of a BS Cum Laude in Zoology from NCSU. This included chemistry, biochemistry, ecology and statistics coursework, as well as graduate level coursework in Toxicology.

Experience:

Mr. McConney's work experience at CompuChem® includes six months in the extraction laboratory performing a variety of extraction procedures including dioxin, pesticide/PCB and acid/base-neutral. Following the extraction lab, he worked for two and one half years in the GC/MS laboratory as an operator, where he performed a variety of analyses including dioxin, volatile, acid, base-neutral and semivolatile. Mr. McConney's responsibilities included limited instrument maintenance and some data review, as well as performing the analysis.

Prior to joining Computher Mr. McConney worked in the Quality Control laboratory of a major pharmaceutical manufacturer.

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Cynthia E. Edwards Final Technical Reviewer CompuChem® Laboratories

Responsibilities:

Presently, Ms. Edwards is a Final Technical Reviewer in the Technical Review Department. She is responsible for the final technical review of commercial samples, organic and inorganic platinum and EPA cases such that adherence to contract protocols and internal quality guidelines are met. Ms. Edwards is also responsible for resolving issues/incidents noted in the review process with the appropriate lab; consulting with the technical and/or quality director/manager to ensure internal quality of deliverable data; documenting and tracking the issues/incidents noted in the review process to communicate with lab managers; and interpreting current contract requirements and current statements of work documents.

Education:

Ms. Edwards received a B.Sc degree in Chemistry and Biochemistry from Spelman College in Atlanta, Georgia. She completed two years of graduate studies in Analytical Chemistry at the University of North Carolina at Chapel Hill. Ms. Edwards has also completed additional course work at Georgia Institute of Technology in Atlanta, Georgia.

Ms. Edwards honors, awards, and significant achievements are as follows: Outstanding Young Women of America, American Chemical Society, Teaching Fellowship in Chemistry (UNC-CH), Dean's List, The National Dean's List, Who's Who Among Students in American Universities and Colleges, Stanford University Linear Acceleration Program (Stanford, California), and Honors Research Program.

Experience:

Experience cont'd.:

ML/LC, melting/boiling point determinations, IR, UV/Vis, NMR, and instrumentation trouble-shooting. Ms. Edwards was also responsible for dose formulation analyses which included method development/validation, recovery studies, homogeneity and stability studies. Technical report preparation was also her responsibility.

Ms. Edwards had completed internships at the following locations: Burroughs Wellcome (RTP, NC), Monsanto Company (St. Louis, MO), Woods Hole Oceanographic Institution (Woods Hole, MA), Ebasco Company and Consolidated Edison of New York (NY, NY), Science Research Institute and Georgia Institute of Technology (Atlanta, GA). She had also worked as a Teaching Assistant for the University of North Carolina at Chapel Hill.

Toney C. Spruell
Final Technical Reviewer
CompuChem® Laboratories

Responsibilities:

In 1988 Mr. Spruell became a Final Technical Reviewer, where he is responsible for the review of all analytical samples and he sees that they meet the prescribed laboratory protocols.

Education:

Mr. Spruell received training in Biology from the University of Norh Carolina from 1975 - 1978 and training from Durham Technical Institute in Engineering from 1979 - 1980.

Experience:

From May, 1978 to August 1983, Mr. Spruell was employed with Monsanto as a Chemical Engineering Research Technician, responsible for research and development on the hollow fiber membrane project (Prism Separators). Mr. Spruell operated and maintained simulations pilot operations using GC and GC/MS as analytical tools to study the flow of environmental gases through these membranes.

Mr. Spruell joined CompuChem® in August, 1983 and has held the positions of GC/MS Trainee, GC/MS Operator and Senior GC/MS Operator where he was responsible for the analysis of environmental volatile samples utilizing GC/MS and for the generation of complete data packages in an accurate and timely manner. This position also served as a Technical Advisor to other GC/MS Operators and Trainees on a particular shift.

Rebecca E. Linvill Final Technical Reviewer CompuChem® Laboratories

Responsibility:

On November 30, 1987 Ms. Linvill became a Final Technical Reviewer, in the Technical Review Department where she reviews commercial and EPA organic lab reports for accurate interpretation of raw data, adherance to internal quality guidelines, and completeness of data.

Education:

Ms. Linvill received a B.S. degree in Soil and Water Science from the University of California, Davis.

Experience:

Prior to joining CompuChem® Ms. Linvill was employed by EMCON Assoc., in San Jose, CA as an Environmental Sampling Coordinator. Her responsibilities were to coordinate environmental sampling team's activities involved in sampling soil, water, wastewater, and sludge for commercial clients.

Ms. Linvill was involved in the interpretation of analytical testing and she recommended monitoring programs. Ms. Linvill also wrote proposals and bids for the Chemical Services Department, and she coordinated distribution and reviewed analytical data for completeness. Field work included monitoring all forms of environmental media.

From 6/84 - 1/85 Ms. Linvill was a Student Assistant, at the State Water Resources Control Board; Sacramento, CA, where she researched the acute, chronic and bioaccumulative effects of trace metals on aquatic organisms. She summarized results for six metals and included an extensive literature search. The project was related to Kesterson Reservoir and the San Luis Drain Research. Ms. Linvill developed water quality criteria using the Kaplow Method for six trace metals.

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Experience cont'd.:

From 9/79 - 6/84 Ms. Linvill was employed as a Lab Assistant at Land, Air, and Water Resources; U.C. Davis, CA. She analyzed soil, water, and plant samples for inorganic constituents utilizing the AA, colorimeter, pH, and EC meters. Ms. Linvill researched the pesticide DBCP and summarized known groundwater contamination.

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## Anna Feather Final Technical Reviewer CompuChem® Laboratories

Responsibility:

In March 1988, Ms. Feather accepted the position of Final Technical Reviewer being responsible for reviewing EPA and commercial inorganic data.

Education:

Ms. Feather received a B.S. degree in biology in May 1986 with minor courses in chemistry at Gardner-Webb College in Boiling Springs, North Carolina.

Experience:

Ms. Feather became a Forensic Drug Testing Screening Technician with the responsibility of RIA, EMIT, and TLC screening for drugs, after one (1) year as a Laboratory Chromatographer, at CompuChem®.

Ms. Feather began her career at CompuChem® Laboratories as a Laboratory Chromatographer in the High Hazard Laboratory, where she was responsible for extracting herbicides, pesticides, and dioxins from soil and water samples.

John C. Tzavaras Developmental Chemist II CompuChem® Laboratories

Responsibilities:

As Developmental Chemist II, Mr. Tzavaras has been responsible for the training of all lab individuals in the preparation and analyses of samples of all types for the determination of metals, cyanide, phenols and any other inorganic constituent using instrumentation available in the inorganics laboratory. He is also responsible for the review of data from a technical quality standpoint.

Education:

Mr. Tzavaras received an undergraduate degree in Chemistry from Tufts University in 1976 and an A.A.S. degree from Durham Technical Institute in Electronics Engineering Technology in 1985.

Experience:

From 1977 - 1980 Mr. Tzavaras was employed by Instrumentation Laboratory as a Product Specialist. From 1976 - 1977. Mr. Tzavaras was employed by Herbert V. Shuster, Inc. as an Analytical Chemist.

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And The

SOPs For The Preparation, Analysis, And
Data Assessment Of Environmental Samples

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## Standard Operating Procedures

**Environmental** 

CompuChem® Laboratories
P. O. Box 12652

3308 Chapel Hill/Nelson Highway
Research Triangle Park

N.C. 27709

Copy Number:

Issued To:

Date:

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Date:

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Standard Operating Procedures

For

Production Planning and Control

CompuChem Laboratories

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For

Glassware Preparation

CompuChem® Laboratories

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For

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CompuChem® Laboratories

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For

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CompuChem® Laboratories

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For

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Standard Operating Procedures

For The

Preparation, Analysis and Data Assessment

of

Environmental Samples

Prepared by the Staff of CompuChem Laboratories, Inc.

Approved by
Robert E. Meierer, Director of Quality Assurance

Copy Number: Approved For Release By: Issued To:

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- i. Instrument Procedure 710 "Acids Lower Detection Limits & Library Search."
- j. Instrument Procedure 711 "Base/Neutral Lower Detection Limits & Library Search."
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- a. Instrument Procedure 101 "Solid or Liquid Pesticide/PCB Analysis."
- b. Instrument Procedure 103 "Solid or Liquid Herbicide Analysis."
- c. Instrument Procedure 111 "As Received Extract Pesticide/PCB Analysis."

#### 4. Metals

- a. Instrument Procedure 001 "Determination of Metals By Flame/Furance Atomic Absorption Spectrophotometer."
- b. Instrument Procedure 005 "Determination of Mercury in Liquid Samples and Digestates from Soils/Sediments/Sludges (Automated Cold Vapor Technique)."
- c. Instrument Procedure 308 "Determination of Metals by Inductively Coupled Plasma."

#### 5. Other Parameters

- a. Instrument Procedure 501 "Determination of Cyanide, Total in Liquid Samples."\*
- b. Instrument Procedure 502 "Determination of Phenolics, Total Recoverable in Liquid Samples."\*
- c. Instrument Procedure 551 "Spectrophotometric Measurement of Formaldehyde."

\*Applicable for analysis of aqueous distillates from manual distillation of 23 solids or liquids.

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#### V. QA/QC Policies

- A. QC Assessment-Semivolatiles, Acids, Base/Neutrals
- B. QC Assessment-Volatiles
- C. QC Assessment-Pesticides
- D. Surrogate Control Limits Liquids/Solids
- E. Liquid QC Spike Acceptance Criteria
- F. Solid QC Spike Acceptance Criteria
- G. Metals/Cyanide/Phenols (Colorimetric) QC Acceptance Criteria
- H. Holding Time Requirements
- I. Compound Lists
- J. Surrogate Standards
- K. Internal Standards

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APPENDIX D

CONDITION CODES

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Appendix D

# Condition Codes

CompuChem® Laboratories uses condition codes to signify either the cause of a sample fraction's failure or the final status of a sample before release (see page 4). The "comments" here describe the consequences of a condition code, the type of analysis for which the code applies, and/or special instructions for using the code. These codes are entered in the Computerized Laboratory Management System (CLMS) under the "COND" column of the "Sample Detail" database and govern the release of the report to the client.

This code list is divided into three sections. The first group of codes are "failure" codes; they apply to all samples repeated because certain criteria have not been met. The codes appear in the Prior (P) slots of the Sample Detail. The next group of codes are "Final" codes used for production samples that have met criteria and may be reported to the client: reports for samples having these condition codes may or may not include the standard Quality

Assurance Notices supplied to each laboratory station. The third group of codes are for Quality Control samples: part A is composed of codes also used for production samples; part B lists codes that apply to quality control samples only. This final list covers Quality Control data that do not meet all Quality Control criteria but are "salvageable" by Quality Assurance if the associated production samples are not affected. Codes from groups II and III appear exclusively in the final (F) slots of the Sample Detail.

At the end of this section is a chronicle of the changes in the Condition Codes over the last year. It is critical to the laboratory system that Dall the most recently revised list be used in each department. This chronicle also ser-

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ves to resolve misinterpretations and misuses of the codes and to explain the applications of the codes further.

The individual laboratory stations are responsible for assigning codes to all issued paperwork, even if no injection is made. As well, every scheduling detail must have an assigned Condition Code. Any questions concerning Condition Codes are addressed to the Senior Quality Assurance Analyst, who monitors the codes periodically to ensure correct application and to pinpoint problem trends for management.

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CODES

### COMMENTS

	OOTE (EXTO
•	CTION AND QUALITY CONTROL SAMPLES
AH = acid surrogates high	Verify vial volume and I.S. areas.
AL = acid surrogates low	Use only when not a chromatography related problem (PC).
BB = bad associated blank	Use for samples which are not analyzed because associated with a bad blanksee chronicle
BF = blank requires florisil cleanup	Used when associated pesticides flori- silled
BH = base/neutral surrogates high	See AH code
BL = base/neutral surrogates low	See AL code
BS = bad associated spike	_Use for samples reprepared due to bad associated sample spike
BU = back-up extract; screened medium	Extracted low level, but not run
CA = cancelled	Applies to all samples (including Quality Control's) cancelled and never analyzed (fill out form)
CL = needs secondary cleanup .	TCDDs needing alumina cleanup
CO = concentration required	Vial volume above mark
<pre>CS = carryover suspected from     previous analysis</pre>	Reinject if rest of data is acceptable see chronicle
CT = contamination suspected	Applies only to effected samples in which contamination is verified
DI = requires dilution	GC/MS usually dilutes sample but may want sample reextracted using less raw sample
DW = wrong dilution used	Lab must rerun at correct dilution AR300730

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ED = extract went to dryness	Usually reextract •
FH = 2-fluorophenol high only	Must re-extract unless I.S. problem
FL = 2-fluorophenol low only	Same as above; verify all areas
FO = foamed during purging	VOA's, reprep at dilution, repurge
<pre>IF = instrument failure; data lost</pre>	Describe failure in comments
<pre>IH = internal standard(s) high</pre>	Reinject unless I.S. solution added; also see IL
<pre>IL = internal standard(s) low</pre>	If extract standard not appropriate, reinject or reextract
<pre>IM = internal standard(s) missing</pre>	Solution not added during preparation
IR = ion ratios outside range	TCDDs
IW = wrong instrument	Injected on wrong OWA - reinject
<pre>JS = reinjection matches previous     analysis</pre>	Use if data fails for same reason; see chronicle
LA = lab accident; sample data lost	Describe LA in comments section
LS = screened low, but really high level	GC/MS run indicates medium level
MS = screened med, but really clean	GC/MS results indicate low level
NM = no match to prior run or duplicate	Applies to appearance of sample extracts or RICs, not % recoveries
OT = other	Describe failure in comments

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OW = wrong original used for Quality Control sample	Automatic re-extraction -
PC = poor chromatography	Perform maintenance if necessary
RI = recovery indeterminate	TCDDs
RN = re-analyzed neat; was run diluted	Used for relatively clean RIC
RO = signal-to-noise ratio out	TCDDs
RU = repeated unnecessarily	An acceptable prior run exists; see Chronicle
SF = Spike recoveries failed	See SOPs for approval criteria
SH = Surrogate(s) uniformly high	See AH, AL codes and chronicle
SI = Spiked Inadvertently	Automatic reextraction
SL = Surrogate(s) uniformly low	See AH, AL codes and chronicle
<pre>SM = surrogate or spike standard     missing</pre>	Solution not added inadvertently
SW = Wrong standard(s) used	Usually automatic reextraction
<pre>UP = unacceptable precision between    QCs</pre>	For comparing SSs or Duplicates (RPDs between spikes, hits, surrs.)
VC = purge vessel cracked	VOAs' reprep sample and repurge
VR = verify results	Sample repeated to verify hits, etc.

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#### II. FINAL CODES FOR PRODUCTION SAMPLES: DATA TO BE REPORTED

DA = dilution acceptable Sample required reanalysis as a dilution; criteria met/qualified EA°= reextraction data acceptable For sample reextracted at least once. even if also reinjected EB°= reextraction data billable Recovery is within +/- 5% of the failing surrogate's recovery ES°= reextraction same as prior QAN required; "matrix" effects confirmed; extraction all data comparable JA°= reinject data acceptable For sample only extracted once and reinjected successfully NS = no sample left for reextraction QAN required; lab responsible for determining deliverability of data OK°= data acceptable first time Never use for repeat status; first injection acceptable as is through RP = reportable prior run Edit failure code to RP if run is reportable; (see Chronicle)

#### III. FINAL CODES FOR QUALITY CONTROL SAMPLES\*

Quality Controls that meet criteria or require laboratory-supplied qualifier:

<pre>AN = quality control acceptable but not reported</pre>	Blanks and blank spikes tripped by syste and run but not needed
<pre>CA = quality control cancelled and   not reported</pre>	All samples (includes Quality Controls) cancelled and never analyzed (fill out form)
DA = dilution acceptable	Quality Control required rerun as dilution; criteria met/qualified by lab
EA°= reextraction data acceptable	For Quality Control sample reextracted; all criteria met/qualified by lab

These are the codes for runs which have valid surrogate data to be entered in 303 the system and used for updating surrogate control limits.

<sup>\*</sup>These are the only codes that will allow associated production samples i "blast" into Phase II. All other codes will hold samples in Phase I

EBo= reextraction data billable	Recovery is within +/- 5% of the failing surrogate's recovery
JA°= reinjection data acceptable	Quality Control reinjected; all criteria met/qualified by lab
OK°= data acceptable first time through	First injection of first Quality Control extract; met/qualified by lab
RP = reportable prior run	Edit failure code to RP if run is reportable; see Chronicle
UN = Quality Control unacceptable but not used	Blanks and blank spikes tripped and run but not needed (See AN code)

Quality Controls that don't meet criteria and/or require special Quality Assurance intervention (Quality Assurance approval or Quality Assurance supplied qualifier) for production sample release:

DQ = Quality Control required dilution and qualified	Not acceptable unless Quality Assurance approves or inserts special Notice
<pre>EQ = Quality Control reextracted     and qualified</pre>	Not acceptable unless Quality Assurance approves or inserts special Notice
<pre>JQ = Quality Control reinjected and qualified</pre>	Not acceptable unless Quality Assurance approves or inserts special Notice
NQ = No sample left for reextraction of Quality Control	Not acceptable unless Quality Assurance approves or inserts special Notice
<pre>0Q = Quality Control is OK and qualified</pre>	Not acceptable unless Quality Assurance approves or inserts special Notice (see Chronicle)

<sup>\*</sup>These are the codes for runs which have valid surrogate data to be entered into the sysem and used for updating surrogate control limits.

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<sup>\*</sup>These are the only codes that will allow associated production samples to "blast" into Phase II. All other codes will hold samples in Phase I.

#### Chronicle of Revisions

Revised 1-31-84: New UP code (note above); also note that EA code is now being used where RA was used--there is no longer an RA code.

Revised 2-5-84: New JS and DA codes. These will prevent the unnecessary counting of problems existing in the first injection and confirmed in the reinject, and differentiate those samples requiring dilution and reinjection from those which had other problems requiring simple reinjection.

Revised 3-28-84: New ES and NS codes. These take the place of the old codes QA and QN, respectively. The purpose was to create codes which would avoid confusion incurred with the old codes. See definitions above.

Revised 7-27-84: Added codes BF and NM.

BF = pesticide blank requires florisil cleanup (since associated samples did)

NM = did not match previous run(s), mate or original (in terms of the appearance of the RIC or chromatogram)

Revised 8-28-84: Added NA code. This code is used for samples which failed but did not require repeating (these will almost always be Quality Control samples). This applies to blanks which fail (can't be reextracted) or sample spikes for which the original failed and was confirmed in the same way (ES).

#### Revised 1-23-85:

#### Codes no longer used --

NR = (Not Required). Use the RU code if repeated in error, or the AN or UN code if its a Quality Control that was run but not needed. Use CA code if it's a cancelled Quality Control that was never run (see CA code below).

NA = (No further Analysis needed). Was being used for Quality Controls that failed but could not be repeated. The EQ, JQ, and DQ codes have been added for this purpose.

DL = (DBC recovery low). Use SL, SH codes for the pesticide and herbicide surrogates.

EM = (Extract Missing). Obsolete.

AR300735

PM = (Paperwork Missing). Obsolete.

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- BW = (Bob Whitehead's area). Was used for tracking purposes, but will have QA Queue now.
- GB = (Lab Go-Back). Used for tracking, but obsolete.

#### Codes added to list --

- EQ = needed to qualify Quality Control data that didn't meet all criteria, but couldn't or shouldn't be repeated.
  - DQ = same as above.
  - JQ = same as above.
- AN/UN = breakdown of old NR code; needed to determine how often Quality Control samples are run unnecessarily and whether or not they passed.
  - CA = same as above, but for those Quality Controls never run; must complete a System Cancellation Form and submit to Scheduling and Control.
  - RU = needed to track repeat request errors and repeats not actually needed.
  - SE = for semivolatile and volatile screens which were not covered in old contract.
- TH/TL/EL/EH = New Caucus surrogates which are no longer advisory.

## Changes/clarifications of existing codes --

- JS = cannot be used as an acceptable final code; always must be used to imply repeat is necessary. For example, if reinjecting to see if peaks are result of carryover, and reinject looks exactly the same, use JA or JQ codes, not JS (even though results are the "same").
- DA = when applying to pesticide data, only use for those samples that require rerun as dilution.
- BB = use only for samples (not blanks) associated with, and never run because of, a bad blank.
- CT = use for contaminated blanks that affect whole batches, or samples that were individually contaminated when the 736 blanks were acceptable; also, when verifying carryover in blanks by reinjecting, and the peaks are actually found to be contaminants present in the extract, change prior CS code to CT.

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- CS = use only when carryover has been verified by reinjection
   (see CT code above).
- NS = individual lab stations are now responsible for determining the validity and "deliverability" of any existing data; a Quality Assurance Notice must be inserted by the lab.
- NM = use the UP code when RPD values fail, and the NM code
   when the possiblity exists that 1) different samples were
   used to prepare duplicates; or 2) the repeat of a prior
   extraction/preparation may have been prepared from a different sample.
- SH/SL = applies also to pesticides and herbicides now, instead of the DL code, which was redundant.

#### Revised 3-28-85:

Code deleted from system --

SE = screen error; this code now subdivided into several more specific codes

#### Codes added to system --

- BU = backup sample; the sample was extracted low level, but is not needed at this time because screen indicates Medium Level. This code is needed for the Low Level extraction queue.
  - FS = screen failure; the blank or blank/spike in the batch screened Medium Level, or the original used for spikes screened Medium and spikes must be reextracted as a result.
  - IW = sample or blank injected on wrong instrument and must be reinjected.
  - LS = screened low, but GC/MS results indicated Medium Level is more appropriate.
  - MS = screened Medium, but GC/MS results indicated Low Level is more appropriate.
  - OQ = Quality Control is OK (first attempt), but needs to be qualified with Quality Assurance Notice or Anaeas Quality Assurance approval.

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RA = RIC appearance is unacceptable; pertains usually to high baseline, large solvent peaks, etc.

# Revised 5-14-85: Codes added to system --

- BS = used when the sample spike(s) and blank spike failed; the entire batch is reextracted and all associated samples get the BS code; the spikes are assigned the normal failure code.
- EB = reextraction results Pass, but one or more surrogate recoveries are within +/- 5% from the failing surrogate's recovery (same as ES, but data passes).
- NF = the final injection is not the one being reported; for a previous run which is later found to pass or is qualified so it can be reported; this code will appear in a "P" slot, even though it was run last.
- RP = reportable previous injection; used in conjunction with NF; the failure code once assigned to this run must be edited to RP on the paperwork and in system. This will be the code appearing in the "F" slot.

#### Revised 1-9-86:

Codes added to system --

- VR = used when sample repeated to verify hits or peaks found in run (particularly for pesticide confirm).
- OM = original screened medium, QC needs to be repeated.
- OL = original screened low, QC needs to be repeated.
- RB = report both runs; use when EPA blank is run on two different instruments but both runs are reportable.

#### Codes deleted from system --

DH = obsolete

EH, EL, PH, PL, Th, TL, NH, NL, YH, YL = specific surrogate failures will be tracked using recovery QUIZ reports. Just use SL, SH, AL, AH, BL, BH codes for surr. failures.

FS = OM and OL will be used in most cases.

NF = will use VR instead; this is a prior code even thoughbits the final run.

RA = obsolete, most should be PC in most cases.

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APPENDIX E

CHAIN-OF-CUSTODY .

AR300739

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Chain-of-Custody

Sample Receiving and Handling:

Depending on the client's requirements, chain-of-custody can be initiated by CompuChem® when the shipping containers (SampleSavers) are sent to the field or by the client at the time of sampling. Custody tape is provided to ensure the integrity of the SampleSaver® and its contents. The chain-of-custody form (see Example 1) accompanying the incoming samples is evaluated and reviewed by the Sample Receiving Supervisor to ensure that document control information is accurate and complete. If samples are not in good condition (i.e. broken or leaking bottles) or chain-of-custody information is incorrect or inadequate, the client is contacted immediately. The condition of the sample including integrity of seals is also noted on receiving documents.

If chain-of-custody is intact, as-received samples are logged into the Computerized Laboratory Management System (CLMS) and scheduled for preparation/ analysis according to the client's analytical requirements. At this point the client's sample identifier is assigned a unique CompuChem® identification number. Labels are automatically generated by the computerized system, and securely affixed to the sample container. The sample is now ready to be transferred to the raw sample storage refrigerator. A copy of the chain-of-custody and sample receiving documents are inserted into a file folder, labeled with the sample's CompuChem® number, and transferred to the Production Planning and Control data files. The original chain-of-custody is mailed to the client with a letter acknowledging receipt of the samples.

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#### Raw Sample Storage:

The Sample Custodian is responsible for organizing and maintaining the security of the raw sample storage refrigerator. Routine access to this locked refrigerator is restricted to the Sample Custodian. Sample containers are removed by the Custodian only when accompanied by the appropriate chain-of-custody tracking forms.

#### Transfers for Sample Preparation:

The Sample Request Form (see Example 2) is used by the individual laboratory stations to request release of raw sample containers by the Sample Custodian. It documents transfer of sample containers from the storage refrigerator to the designated sample preparation laboratories.

#### Transfers of Prepared Samples to Storage:

Once the extract/aliquot is prepared from the raw sample, it is returned to the Sample Custodian for storage. The Extraction Worksheet (see Example 3A) or Sample Preparation Worksheet (see Example 3B), depending on the preparation requirements, is used to document this transfer. Again, the prepared samples are stored in a locked, restricted-access refrigerator.

#### Transfers of Extracts to Instrument Laboratories:

Depending on the analytical requirements, the sample extract is released to the appropriate instrument lab. Chain-of-custody for this transfer is documented by any of several lab Worklists, divided according to fraction type (see Examples 4A, 4B, 4C, 4D and 4E). Pesticide and TCDD extracts, because they can be analyzed in large numbers via autosampler sequences, are sent directly from the

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Sample Preparation Laboratory to the GC Laboratory. Chain-of-custody for this transfer is accomplished with the use of the appropriate Extraction Worksheet (see Examples 5A and 5B).

Transfer of Extracts from Instrument Laboratories to Sample Storage:

After the laboratories have completed the sample's scheduled analysis, the extracts are returned to the sample storage area. The appropriate lab worksheets are signed by the Sample Custodian and the individual returning the sample. The sample is then stored in the appropriate storage refrigerator.

Commercial samples are stored for thirty days after reporting, and EPA samples are stored for 60 days after reporting on the analyses. Sample extracts are stored for 6 months. When these storage periods have expired, the samples and extracts are disposed of as hazardous wastes, in accordance with Federal and State regulations.

## Data Report Chain-of-Custody:

Computer-generated hardcopies from the instrument analysis contain the sample identification number on each page. As part of the data report, the GC or GC/MS Worksheets (see Examples 6A and 6B) are used to record information pertinent to the analysis of each extract (i.e. instrument, data of injection, analyst, etc.). Once the data report is assembled and evaluated by the laboratory's Data Review Staff, it is transferred to the Production Planning and Control data filefolder. Along with the original chain-of-custody and sample receiving information, the report is reviewed by a member of the Technical AR 300742 Review Staff. A key concern in this step of the review hierarchy is to ensure that the chain-of-custody is documented and unbroken.

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# Data Storage:

Copies of the data report and all associated chain-of-custody documents are archived in a locked, off-site storage facility for an indefinite period of time.

COMPUCHEM LABORATORIES

# EXTRACTIONS AND VOLATILE SAMPLE REQUEST FORM

			Laboratory:	
EPA	Water	· · · · · · · · · · · · · · · · · · ·	Requested By:	
Comm.	Soil	<u> </u>	Date:	
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# INORGANICS SAMPLE REQUEST FORM

ECK WHERE APPL	ICABLE:	. –	Laboratory:			
EPA	Water					
Comm.	Soil					
CompuChem #	Pulled ( )		Samples for 1 2	- 3 shift		
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ASSIGNED 10. --SPINE SWEWOOME SAMPLE NUMBER <u>5</u> **₹** ₹ ē <u>₹</u> 8 CODE PART S-Val - C E ₽ EPA \* E OC SAMPLE 왕 Pust VOLUME (IIII) SCHEEN SAMPLE TCDD FUNAL EXTRIACT VOT (MIS) Other S ACE A EXTRACTS RECEIVED BY FINAL VOLUME VERIFIED MANUAL COUNTER \_ SUPERVISOR REVIEWED PEST B/X ADJUSTED PH > COMPT DATE ASSIGNED PACE AR3002478 Ş AR300747

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)27

#### VOLATILE PREP WORKSHEET

			Sample QC Sample Weight (g) Date			!						
Sample Number	Prep Code	Case No.	Type	Sample Origina		ht (g) me (mi)	Dete Comp.	LIQ	Screens Q S L N		Comments	
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\_\_\_\_\_Date \_\_\_\_\_

GC/MS	WORKLIST	
SAMPLE	REQUEST	FORM

	CASE	
DHE	DATE	
DUL	DAIL	

## DELIVERABLES CODE/INSTRUMENT CODE

CompuChem #	Sample Prep	Date Run	OWA#	Operator	Date Reviewed	<b>Com</b> ments
1.						Blank <b>#</b> 1
2.						Blank # 2
3.						Sample Spike
4.						Sample Spike
5.		<u></u>				Original Used
6.						
7.						
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Appendix F
Revision No. 0
Date: November 16, 1987
Page 1 of 4

#### APPENDIX F

#### Drinking Water Requirements

Samples identified by the client as "Drinking Water Samples" (i.e., for drinking water compliance monitoring) require certain special handling and reporting procedures, but are otherwise handled by the Computerized Laboratory Management System (CLMS) in much the same way as non-compliance samples.

The Sales Representative in the Marketing Department is responsible for placing the order in the CLMS, ensuring that the appropriate analysis codes are chosen. Only analysis codes describing EPA-approved drinking water methods may be used. The tables on the following pages identify the particular methodologies utilized in processing drinking water samples.

For compliance monitoring in North Carolina, following the "Rules Governing blic Water Supplies" (amended February 1, 1987), all certified commercial boratories are required to report results of analyses to both the Public Water Supply Branch and the supplier of water (client). The rules specify the particular reporting forms to be used and the time period in which reports are to be submitted.

In evaluating drinking water sample data, the QC criteria applied are as specified in the referenced method. Where unspecified, CompuChem employs those criteria outlined in the Federal Register (October 26, 1984 600-series methods) for "Water and Wastewater", presented in Section 9.5 of the QA Plan. Once a database of sufficient size is generated, control limits for precision and accuracy will be generated based on historical data for aqueous sample analyses.

In order to continue providing analytical services for compliance monitoring, CompuChem must maintain certification through the various drinking/potable water certifying agencies. The North Carolina Department of Human Resources (NCDHR), Division of Health Services, regulates certifications, performance evaluations and annual on-site laboratory inspections for these services in North Carolina. CompuChem also maintains drinking water certifications in a number of other states, many of which accept reciprocal certification through the NCDHR.

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# METHODS USED BY COMPUCHEM FOR POTABLE WATER ANALYSIS

Volatile	Organic Contaminants/THMS	Method Used
	Bromobenzene	524.1
	Bromochloromethane	524.1
<del>-</del>	Bromdichloromethane	524.1
	Bromoform	524.1
	Bromomethane	524.1
	sec-Butylbenzene	524.1
	tert-Butylbenzene	524.1
	Carbon tetrachloride	524.1
	Chlorobenzene	524.1
_	Chlorodibromomethane	524.1
	Chloroethane	524.1
	Chloroform	524.1
	Chloromethane	524.1
	o-Chlorotoluene	524.1
	p-Chlorotoluene	524.1
	1,2-Dibromo3-Chloropropane	504,524.1
	Dibromomethane	524.1
	o-Dichlorobenzene	524.1
	m-Dichlorobenzene	524.1
	p-Dichlorobenzene	524.1
	Dichlorodifluoromethane	524.1
	1,1-Dichloroethane	524.1
	1,2-Dichloroethane	524.1
	1,1-Dichloroethylene	524.1
	cis-1,2-Dichloroethylene	524.1
	trans-1,2-Dichloroethylene	524.1
•	Dichloromethane	524.1
	1,2-Dichloropropane	524.1
	1,3-Dichloropropane	524.1
	2,2-Dichloropropane	524.1
	1,1-Dichloropropane	524.1
	1,3-Dichloropropane	524.1
	Ethylbenzene	524.1
	Ethylenedibromide	504,524.1
	•	- 524 <b>.</b> 1
		524.1
	Isopropylbenzene	524.1

<sup>\* &</sup>quot;Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water", September, 1986, EMSL-CI, U.S.EPA Cincinnati, Ohio 45268.

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#### METHODS USED BY COMPUCHEM FOR POTABLE WATER ANALYSIS (continued)

Volatile Organic Contaminants/THMS	Method Used *
n-Propylbenzene	524.1
Styrene	524.1
1,1,1,2-Tetrachloroethane	524.1
1,1,2,2-Tetrachloroethane	524.1
Tetrachloroethylene	524.1
1,1,1,-Trichloroethane	
	524.1
Trichloroethylene	524.1
Toluene	524.1
1,1,2-Trichloroethane	524.1
1,2,3-Trichloropropane	524.1
Vinyl chloride	524.1
o-Xylene	524.1
m-Xylene	524.1
p-Xylene	524.1
Chlordane Polychlorinated Biphenyls	608 608
Inorganic Contaminants	. Method Used **
•	. 026 0
Iron	236.2
Manganese	243.2
Arsenic	206.2
Barium	
Cadmium	213.2
Chromium	
Fluoride	3005 7
	340.2
Lead	239.2
Lead	239.2 245.1
Lead	239.2

<sup>\* &</sup>quot;Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water", September, 1986, EMSL-CI, U.S.EPA Cincinnati, Ohio 45268.

Silver

Sodium

272.2

273.2

<sup>\*\*&</sup>quot;Methods of Chemical Analysis of Water and Wastes," EPA Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, 45268 (EPA-600/4-79-020), March 1979. Available from ORD Publications, CERI, EPA, (EPA-600/4-79-020), march 19/9. Available 1100 Cincinnati, Ohio, 45268. For approved analytical procedures for APC 19/52 technique applicable to total metals must be used.

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# APPROVED METHODOLOGY FOR ORGANIC CONTAMINANTS

Additional	Organic Contaminants		Method Used
	Chlorinated Hydrocarbons		1
	Endrin		1
	Lindane		1
	Methoxychlor	t.	1
	Toxaphene		1
	Chlorophenoxy, Acids		2
	2,4,-D		2
	2,4,5-T	-	_ <b>2</b>

- 1: "Methods for Organochlorine Pesticides and Chlorophenoxy Acid Herbicides in Drinking Water and Raw Source Water," Available from ORD Publications, CERI, EPA, Cincinnati, Ohio 45268. (pp.1-19)
  - : Ibid. (pp. 20-35)

APPENDIX G
Subcontracted Services

Section: Appendix G

Revision No. 0

Date: October 3, 1988

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#### Subcontracted Services -

Subcontracted services are regulated to comply with the requirements of the Quality Assurance Program. The Marketing Department establishes, with input from the laboratory, when subcontract requirements are needed. The QA Department verifies that the subcontractor complies with the methods written in their referenced SOPs and with their own QA Plan requirements. This is accomplished by an on-site inspection of the subcontractor facility. The same criteria and objectives used during an internal Systems Audit are used for the subcontractor audit. Prior to the approval of a laboratory for its analytical services, blind PE samples are submitted and must be successfully completed as part of their performance audit.

The Director of QA has final authority over the approval of all subcontractor services. The documentation of subcontractor certification is maintained in QA Department files and is made available to clients upon request. Subcontractors are not used when specifically restricted by a client's QAPP, statement-of-work, or contract, and clients are notified whenever a subcontractor is to provide analytical services.

APPENDIX H
Preventive Maintenance

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## COMPUCHEM LABORATORIES

## BRIEF DESCRIPTION OF INSTRUMENTATION SERVICE

THE INSTRUMENTATION SERVICE DEPARTMENT OF COMPUCHEM LABORATORIES PROVIDES A VITAL ELEMENT IN THE ON TIME PRODUCTION OF CUSTOMERS NEEDS BY BEING AN IN HOUSE SERVICE ORGANIZATION. THE INSTRUMENTATION GROUP CONSISTS OF A MANAGER, SIX ELECTRONIC INSTRUMENTATION TECHNICIANS AND A STAFF CONSULTANT. ALL OF THE PERSONNEL HAVE BEEN TRAINED BY REPRESENTATIVES OF THE MANUFACTURER AS WELL AS ON THE JOB TRAINING. WE ALSO WORK CLOSELY WITH THE BUILDING MAINTENANCE STAFF WITH ANY INSTALLATIONS OR CHANGES THAT MUST BE MADE.

THE PRIMARY FUNCTION IS TO PROVIDE IMMEDIATE RESPONSE TO REPAIR NEEDS OF THE GC-MS FLOOR AND PREVENTIVE MAINTENANCE ON A ROUTINE BASIS. THE GC LAB HAS OVER 20 GAS CHROMATOGRAPHS IN PRODUCTION FOR WHICH WE MAINTAIN SUPPORT. THE INORGANIC LABORATORY INSTRUMENTATION IS GENERALLY SERVICED BY OUTSIDE VENDORS UNDER A SERVICE AGREEMENT; HOWEVER WE DO PROVIDE AS MUCH ASSISTANCE AS POSSIBLE IN RESOLVING THEIR EQUIPMENT PROBLEMS.

AN INVENTORY OF APPROXIMATELY \$200,000 IN SPARE PARTS FOR INSTRUMENTATION ENABLES THE COMPANY TO MAINTAIN AN UPTIME OF GREATER THAN 97% FOR THE GAS CHROMATORGRAPH--MASS SPECTORMETER EQUIPMENT WHICH CONSISTS OF 24 INSTRUMENTS FOR ENVIRONMENTAL AND 13 FOR CLINICAL FDT..

WE RUN THREE SHIFTS A DAY 5 DAYS A WEEK WITH A FULL SHIFT ON SATURDAY AND ON CALL COVERAGE DURING OTHER TIMES OF THE WEEKEND.

ALL ASPECTS OF PROVIDING GOOD EQUIPMENT OPERATION IS FOREMOST IN OUR DAY TO DAY OPERATIONS, FROM HAVING ON-SITE BULK GAS SUPPLY SYSTEMS OF HELIUM, LIQUID NITROGEN, AND HOUSE AIR, TO ELVALUATING NEW EQUIPMENT AND METHODS AS WELL AS HAVING NEEDED PARTS WHEN NEEDED.

FEBRUARY 10, 1989

INSTRUMENTATA ON 358 OF 705 HANGER

I. LEE GP

# S.O.P. FOR QUARTERLY PREVENTATIVE MAINTENANCE OF FINNIGAN MODEL OWA MASS SPECTROMETER

# I Servicing Analizer

Go through system status to make sure filament, multiplier, and cal. gas are off. Disconnect cal. gas leads.

Vent instrument putting pump/vent switch to vent. Switch analizer voltage to standby.

Disconnect high voltage, anode, and the two RF leads on back of M.S. flange.

Remove four bolts on front flange with 1/2" ratchet wrench. (Make sure you wait for vacuum to be vented. Do not pry open the flange with a screwdriver.). Also lay front flange down-orented so you can put it back the same way.

Remove rear flange bolts. Do not alow flange to drop down against manifold because it could damage RF feed-through.

Take analizer (rear flange portion) into shop. Remove leads from top of source. Remove source. Unscrew (partially) small screw on side of rod can. Pull rods out (using cloth gloves). Do not drop rods. Clean the inside ends of rods with lapping paper. Rinse with methanol. Blow off with house air (nitrogen). Replace rods. Tighten screw. Replace source. Replace connections. Test for short. Replace analizer back into M.S. manifold. Replace front flange bolts.

# II Turbo Pump

Remove caps from side of turbo pump. Put 'o' rings onto these caps if they do not come off with it. Be careful with small springs, they can come out. Draw-out old oil with syringe. Put oil in a beaker. Replinish with synthetic oil A401D, pulling up cap to release oil through its tube. Fill to about 1/8" away from metal rim inside. Do this on both sides of turbo pump.

Replace cap being careful to place cap center slot onto spring. Then tighten firmly, but do not exert much torque.

# III Rough Pump

Remove clamp from top of rough pumps (separator and fore). Put centering ring aside with clamp. This will disconnect the vacuum hose. Slip up and off blue vent hose.

Unplug pump and carry into shop to change oil. Unscrew top and bottom oil plugs and hold used oil container with funnel to catch oil being drained.

Replace bottom plug and fill pump with new TKO-19 oil to center of view window. Replace the rough pumps in the same mannor as they were removed.

Pump down system : pump/vent switch to pump, analizer voltage switch to ON.

Replace card cage filter 16x16x1. Vacuum under and around instrument. This will include top-back of power drawer and disk drive panel.

## IV Tune RF

After system is pumped down and reset light depressed, go into M-tune. Put First Mass 100, Mass Range 0, Scope Sweep ON. Using a DC voltmeter connected to two test points of the RF Generator, tweek knob to lowest voltage reading. Repeat this step for First Mass of 400,600 and 800. The reading should be between >5 and 1.5 volts D.C.

#### V M-Tune

Check for air leak in Scan L and tune instrument to FC43 to approximate values. Calibrate below 10%. To prove good zero, the processor time should be 25% +/- .5

Disconnect cal. gas solonoid leads and return instrument back to operator.

## VI Record

Record PM on: (1) Service report (2) Yellow PM schedual card (3) Magnetic board.

Date Issued:	· - ·.	Approval:		
Written By: David Rich	,		Mgr	The full of balion

## S.O.P. FOR CLEANING OF SOURCE PARTS

## ----- GENERAL SOURCE CLEANING TIPS -----

- 1) Mix aluminum oxide & water into a paste.
- 2) Clean each part with cotton tip applicator & Dremel. (Dremel set at speed 1 is sufficient.)

- 3) Don't use too much force on any part (not necessary).
  4) Rinse each part cleaned and set in methanol.
  5) Ultrasonically clean all parts afterward for no more than a minute or two.
- 6) Rinse off all parts with water, drain, put in a GC oven at 140 degrees for 10 minutes.
- 7) Bake all ceramics in furnace at 4.5 (% time on) for approximately 2 hours (about 1500 degrees C).

# ----- OWA SOURCES -----

- 1) Be sure all areas of source pieces are cleaned thoroughly.
- 2) Most critical pieces to clean thoroughly are collector and ion volume (face areas especially).
- 3) SS connectors should also be cleaned with screw holes facing correctly when assembling.

## ----- MSD SOURCES -----

Major source parts to be cleaned :

- 1) Repeller face
- 2) Drawout lens face and inside surface
- 3) Ion source chamber (inside and out)
- 4) Any other metal part that looks dirty

Revised: March 8, 1988 Approval: Written By: Ted Silver Mgr. Laboratory Instrumentation

# S.O.P. FOR OWA SOURCE ASSEMBLY

Make sure adapter (source base) is clean and has 4 studs of relatively equal length.

Place aperture on adapter, keeping in mind 2 holes closely situated together on adapter (source base) for correct orientation.

Install 4 ceramic bushings (.360), then 4 sapphires that fit over the bushings.

Place lens next over bushings. Note that the lens and extractor have the same part no. but the lens has a slight indentation at corner. With 2 closely situated holes (on source base) to the left, lens indentation is on right (bottom).

After placing 4 more sapphires, position extractor next with same orientation previously explained (SS connector is on top right).

Place 4 ceramic bushings next (.250), then ion volume. Reeping orientation as previously discussed. (SS connector on ion volume is at top left)

Install 4 more sapphires, then 4 flat washers, then 4 hex nuts.

Position on right (middle) is where collector will go. First place ceramic bushing (.100), then ceramic bushing (30004-20030), then collector, sapphire, washer, and screw (1/4").

Position on left side where 2 closely situated holes are is where filament will sit. Place 2 ceramic bushings (.100), then ceramic bushings (3004-20030), then position filament (with rhenium wire and larger openings facing inside). Next position 2 sapphires, 2 washers and 2 screws (1/4").

Observe ionizer (source assy.). Distance between ion volume and collector should be such that a paper clip can be inserted between them. Filament distance from ion volume should such that SS connectors on filament should not be touching ion volume, and not shorting at any point.

Be sure all ceramic spacers and/or bushings used as pairs are same size.

Do not overtighten any screws. Make sure all parts are seated correctly.

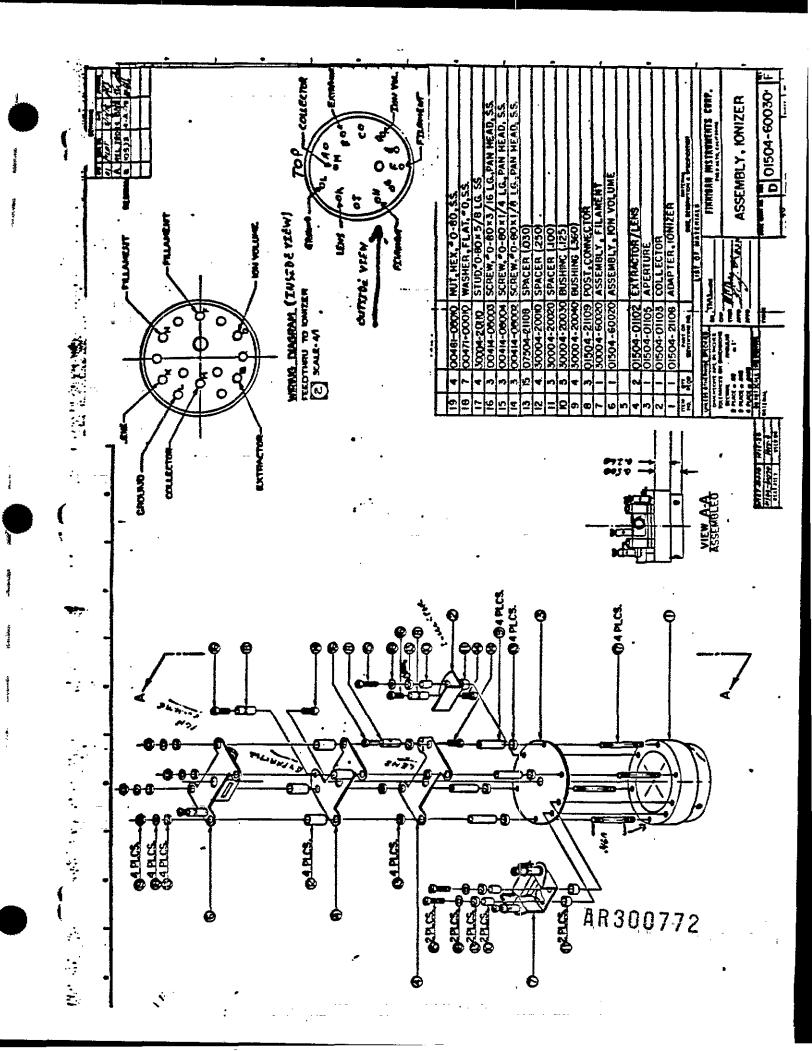
Note: Refer to OWA 1000 series schematics, Section 4-4.

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	Approval:	HILO
	. The second second second second second second second second second second second second second second second	

Date Issued:

Written By: Ted Silver

Mgr. Laboratory Instrumentation



Ţ	]	Hard	COPY	M-Tune	peaks.
---	---	------	------	--------	--------

- [ ] Disconnect cal. gas leads befor venting instrument.
- [ ] Change source and clean rods.
- [ ] Test for shorts across source connections.
- [ ] Inspect 'o' rings on M.S. manifold flange.
- [ ] Change oil in turbo pump.
- [ ] Examin turbo pump wick.
- [ ] Check 'o' rings and springs in turbo pump cap.
- [ ] Change oil in rough pump.
- [ ] Replace vacuum and vent hose on rough pump.
- [ ] Pump down system.
- [ ] Replace card cage filter.
- [ ] Dip R.F. voltage to lowest point.
- [] In M-Tune
  - (A) Check for air leak in scan 'L'
  - (B) Tune instrument
  - (C) Calibrate
- [ ] Disconnect cal. gas leads.
- [ ] Return instrument to operator.
- [ ] Record PM on:
  - (A) Service report
  - (B) Yellow PM schedual card
  - (C) Magnetic board

		****
OWA #	; Date :	AR200770
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Completed by :

#### EC = EPOS

PART NO R5060267.001	Manu Endias	DESC PCF E2/0	COUNT 1.	<u>Value</u> 321	Andunt 321
<b>R5040301.001</b>	EMDIAS	SAMPLE TABLE COMPLETE	1	4220	<b>622</b> 0
R5060338.002	EMDIAG	CHAIN DRIVE PINNEL	2	930	1860
<b>R5060452.000</b>	ENDIAS	TEMP. RING	1	<b>3</b> 63	363
R5060460.002	endia5	BEARING RING	2	480	960
<b>R5060465.004</b>	EMDIAS	ROTOR DRIVE COMPLETE	1	1330	1330
R5060564.000	ENDIAR	RINCEHEAD 3 HOLDER	1	\$20	400
R5060567.009	EMDJAS	R2 CARRIER	1	1950	1950
R5060554.007	EMPTAS	WASTE PUMP	1	541	<b>5</b> 41
R5069556.005	Endias	VACUUM PUNF COMPLETE	3	1310	<b>39</b> 30
R5060712.001	ENDIAS	PCB A/D CONVERTER	1	450	450
R5060726.002	EMDIAS	PCS PROCESSOR	-1	1490	1480
R5060737.004		PCF UPI 0	i	990	650
R5060739.007		PCB UPI 2	1	660	650
R5060750.000	EMDIAS	PCB EPF, INTERFACE	1	1090	<b>109</b> 0
R5060754.006	Embias	POB TEMP. CONTROLLER	1	305	305
R5060756.009	EMDIAB	P/S DIS/EDP/PRINTER	1	410	410
<b>%5</b> 950762.009	EMPIAE	PEB MEU/MIXER P/S	1	392	392
R5060765.008	ENDIAS	PEB AL/DC 1	i	<b>6</b> 30	<b>68</b> 0
<b>R</b> 5060769.003		PCB MCU STEEPER MOTOR	i	760	760
R5060780.007	ENDIAG	PCS MEMORY	i	<b>2</b> 070	2070
	ENDIAG	PCB POWER DN	2	480	960
<b>\$5040810.003</b>	ENDIAG		1	4(10	400
	EMDIAF	PCB PRINTER CONTROL	2	450	<b>70</b> 0

ÄR300774

•				THERMOMETER	i	750	759
Suspery Total	for REC (Count					24912	<b>2</b> 9872

#### $\mathbf{E}\mathbf{E} = \mathbf{F}\mathbf{I}\mathbf{E}$

PART ND 00950-00206	HANU ETHUTGAU	BISMA 3 P/S	COUNT +	<u>VALUE</u> 0900	AMDUNT 0700
		******************	1	U7QU	**********
		TRIP READY PCB	2	550	1100
	FIRRIGAN	TEMP CONT DOB	3	<b>67</b> 5	2025
00950-00412		SIBNA 3A UPPOCESSOR	1	1400	1400
	FINKI64K	SISMAJA UPROCESSOR	2	1400	<b>25</b> 00
)1504-61623	FINNISAN	BUADAPBLE RDDS	1	5900	5000
)1583-60000	FINNISAN	PRE AMP ASH	1	<b>0</b> 850	<b>0</b> 850
00006-20010	FINNISAN	MARJFOLD	1	<b>22</b> 55	2255
10001-21010	FINNISAN	MOTHERPOARD PCB	1 .	1250	1250
6901-60020	FIMRISAN	R.F. BENERATOR	2	3266	<b>65</b> 32
0001-61020	FIRRIBAH	DISTAL 1/D	3	4895.	14685
10091-61830		IDN SOURCE	4	1580	. 6320
19001-81041	FINKIBAN	RDD DRIVER	<i>ž</i>	1450	4350
040141060	FINNISAR	TEMP CONT. PCB	1	1100	1100
10001-61150	FINNIBAN	AUTO-SAMPLER	3	4000	<b>120</b> 06
10002-60020	FINNIGAN	3KV POWER SUPPLY	. 3	2100	<b>63</b> 00
10002-61010	FINNIGAN	VAC INTERLIDEK	2	1600	3200
1 <b>0</b> 002-61020	FIRRIGAN	HEATER TRIAL PCB	1	300	<b>30</b> 0
0002-61030		MS POWER SUPPLY	2	<b>B</b> 75	1750
	FIRNIGAN	BC 1/0 PBC	2	550	1100
0108-04910	FINNISAN	MECH PURF 2002		1210	APR 300775
0108-04500	FINNIEAN	HECH PUMF 2004	2	800	1600

20000-61010	FINNISAN	· · · · · · · · · · · · · · · · · · ·			18000
20011-61001	FINRISAN	DUAL SERIAL 1/D	3 .	<b>25</b> 00	<b>75</b> 00
50010-60016	FINNISAN	FRONT FLANSE	1	1388	1388
EM12001	DETECH	MULTIPLIER	16	<b>4</b> B0	<b>108</b> 80
INCOSSOEM	DETECH	MULTIPLIER	2	780	1560
for REE (Epun		••••••		,	*********
•	• •		<b>7</b> 3	47854	123405

#### BEE = FF

Bussary Total

PAPT ND 03890-60010	MANU MENLETT PACKA	<u>DESC</u> MAIN BOARD	COUNT 1	<u>VALUE</u> 1550	AMDUNT 1550
<b>0</b> 5970-60009	HERLETT PACKA	POWER SUPPLY		<b>75</b> 0	<b>75</b> 0
<b>0</b> 597 <i>6</i> -6005£	MEKLETT PACKA	SDURCE ASSEMBLED	3	1900	<b>3</b> 7 <i>6</i> 0
05970-60114	HEKLETT PACKA	RFPA BDARD.	1	700	700
<b>059E5-6</b> 0301	HEKLETT PAEKA	BARD 1/D	i	410	410
<b>05925-6</b> 0317	HERLETT, PACKA	HISH VOLTASE	1	450	450
<b>0</b> 5990-60005	NEWLETT PACKA	BUAD DRIVER AE	1	1225	1225
0599(-60205	HEWLETT PACKA	ION OPT PCE AS	1	<b>5</b> 00	506
0579(-69416	MEWLETT PACKA	DETECTOR	1	525	525
<b>07673-60</b> 020	HERLETT PACKA	PIB	2	<b>8</b> 50	1700
<b>07</b> 133-67530	HEWLETT PACKA	DISK, CONTROLLER, BOARD	1	<b>3</b> 00	<b>3</b> 00
18594-60060	HENLETT PACKA	CONTROLLER	1	420	420
18594-60070	HEMLETT PACKA	MAINTRAME	1	\$50	550
18596-60520	HEHLETT PACKA	R FLEX	1	<b>3</b> 50	<b>3</b> 50
19242-60010	HENLETT PACKĀ	INET BOARD	1	430	430
#5D\$30 <i>6</i>	DETECH	MULTIPLIER	4	420	2480
	,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,				

Bushary for RED (Doubt = 16): Total

22 15530

BEC = SHOP

FART NO	MANU	DESC	COUNT	VALUE	<u>ambunt</u>
005-13549	DATA GENERAL	MOVA 4 CPU/MEMBRY		\$50	2200
<b>9</b> 03-5850	DATA BENERAL	NOVA 3 CPU	2	450	900
<b>0</b> 05-7787		MDVA 3 MEMORY	•	450	1350
	OFI TWO! BEW		4	<b>3</b> 36	£344
21-150	SLO-NAC	LEAK DETECTOR	2	480	<b>3</b> 70
3/12		MDVA 3/12 COMPUTER	1	1200	1200
4010	TEKTRONIX	TERMINAL	1	1000	1000
465	TEKTRONIX	DSCILLESCOPE	1		<b>2</b> 906
4658	TEKTRONIX	DSCILLDSCOPE	1	3100.	3400
604-3	DRANETZ	LINE ANALYZER	1	2000	2000
<b>6</b> 60 <b>-</b> 2	RODRY HOUNT	SPOT WELDER	i	749	749
		96MB DISC DRIVES		750	1500
<b>B</b> 5-100	KELLEY	DESOLDERING STATION	1	749	74?
<b>K20</b> 050	ACOPIAN	POWER SUPPLY	i	325	325
MDVA 4E	DATA BENERAL	NOVA 41 COMPUTER	2	900	1800
NOVA 4X	DATA BENERAL	MOVA 4% COMPUTER	1	2500	<b>2</b> 560
5%2-12 <u> </u>	MINI COMP TEC	DISC CONTROLLER	2	1300	2600
TEP-270/300		TURBO CONTROLLER	2		3000
TPH-270/330	BALZER	TURBO PUNES	4	<b>3</b> 470	13880
**********	• • • • • • • • • • • • • • • • • • • •	47114447441747744444			**1******
for REC (Count	t = 19}:	•	36	25009	44357
for REPORT (Co	ount = 87);		A.B.	24444	<b>0.07</b>
			162	109305	215674 A D 2 O O 7 T
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Summary Total

OWA #	OWA	TYPE	DATE	s/n
1	3039	1020	9/81	12137-0980
2	<b>3</b> 053	1020B	9/81	12391-3-0281
3	3031	1020	9/81	12141-0980
4	3028	1020	9/81	12138-0980
5	3024	1020	9/81	12140-0980
1 2 3 4 5 6 7	3021	1020	9/81	11957-2-0180
7	3017	1020	9/81	11957-3-0180
8 9	3013	1020	9/81	11957-4-0180
9	3079	1020	9/81	11001-4-0100
10	3042	1020	9/81	11957-1279
11	3046	1020B	9/81	12391-2-0280
12	3067	1020B	9/81	12391 0281
13	3035	1020	9/81	12139-0980
14	3055	1020B	6/82	12391-1-0380
15	3059	1020B	9/81	12391-4-0381
16	3063	1020B	9/81	12391-5-0381
18	2314	1020B	6/83	12645-1-1181
19	2311	1020B	6/83	12645-4-1181
20	2318	1020B	6/83	12645-6-1281
21	2322	1020B	6/83	
22	2304	1020B	6/83	12645-3-1181
23	2307	1020B	6/83	12645-2-1181
		7050D		S12645-5-1281
INCOS 50			1987	13954-0387

# GAS CHROMATOGRAPH LABORATORY EQUIPMENT

MODEL #		SERIAL #	CCHEM #	<b>A-</b> D	TYPE	Installation Date
VARIAN VARIAN VARIAN VARIAN	3700 3700	58760308-13 71280469-13 32968966-11 74550509-13	000000	2&3 7&1 23	DUAL ECD AUTOSAMPLER DUAL ECD AUTOSAMPLER FID NPD FID	MAR 1980 NOV 1980 JAN 1980 JAN 1982
HP 5886	D	2236A04163		21 .	FID	AUG 1982
VARIAN VARIAN VARIAN VARIAN	3400 3400 3400	2006 2310 2309 2312	001177 001175 001178 001173	5 0 4	FPD RCD NPD AUTOSAMPLES ECD NPD AUTOSAMPLES ECD FID AUTOSAMPLES	R 1986 R 1986
VARIAN VARIAN VARIAN VARIAN VARIAN	3400 3400 3400	3623 3052 2308 2307 2311	001174 001179	9 10 12 14 24	ECD FID AUTOSAMPLEI ECD AUTOSAMPLEI ECD AUTOSAMPLEI ECD AUTOSAMPLEI ECD AUTOSAMPLEI	R 1986 R 1986 R 1986
VARIAN TEKMAR TEKMAR O.I.	LSC-2	3053 144 1016 6411-6-155	001357 001647	19	HALL DET PURGE AND TRAP AUTOSAMPLER	1985
VARIAN O.I. HNU	3400 4460 PI-52	3054 171-6-9B 620045	001356 001499 001362	20	PID DET PURGE AND TRAP	1985
VARIAN TEKMAR TEKMAR HNU	LSC-2	2306 1821 1041 620100	001176 001241 001648	18	PID PURGE AND TRAP AUTOSAMPLER	1985
VARIAN TERMAR TERMAR O.I.	LSC-2	2005 1556 902 6644-5-102	000953 001316 001649	17	HALL PURGE AND TRAP AUTOSAMPLER	1985
VARIAN		3055 521-6051C 365-6-0020	001358 001507 001508 001509	16	PID PURGE AND TRAP LOOP SAMPLING MODULE	1985
BLUE M	SW-11T	A-1. SW365	001353		OVEN	
HP 335	7				ALS SYSTEM DATA PROCESSING	
HP 335'	7				ALS SYSTEM AR300 DATA PROCESSING	779.

CHARCOAL AIR FILTERING SYSTEM

# INORGANIC LABORATORY INSTRUMENTATION

IARE	MODEL	SERIAL #	INSTALLED
ECHNICON	TRAACS 800		1987
CIENTIFIC PRODUCTS	SPZ-410	21116	
RECISION	CIRCULATING BA	TH	
ALANCE METTLER	MODEL HL 52	A76373	
APRELL ASH "I C P"	MODEL 1100	:	5-30-86
ICROPROCESSOR IONALYZER PH METER	901	93353	11-79
ARIAN CARY	219	0438812	01-81
ECHNICON CYANIDE/PHENOL AUTOANALY	ZER		
AUTOSAMPLER		PR3357	
PUMP		GG0797940	
MANIFOLD CYANIDE		C60164	
MANIFOLD PHENOL	. ·	TC60222	
S.C. COLORIMETER		PR1432	
PRINTER		78TR/65920	
TEMP BATH			
NSTRUMENTATION LABORATORY AAS	VIDEO 12	INSTALLED	04-16-86
AA .	857	2128	
USED WITH I L	AVA 440	1625	

# INURGANIC LABORATORY INSTRUMENTATION

INSTRUMENTATION LABORATURY AAS	VIDEO 22	INSTALLED 02-05-86
<b>A</b> A	857	2127
FASTAC	254	2475
FURNANCE	655	3471
COOLANT CIRCULATOF	HOUSE WATER	1265
AUTOSAMPLER	254	136510
VACCUM PUMP		<b>-25</b> 3257
EDL POWER SUPPLY WESTINGHOUSE	185	A7935466
EDL POWER SUPPLY WESTINGHOUSE	185	A8017483
PRINTER	4528-T	920382
LABORATORY INSTRUMENTATION AAS	VIDEO 22	INSTALLED
AA	857	
FASTAC	254	2027
FURANCE	655	2961
COOLANT CIRCULATOF	HOUSE WATER	
AUTOSAMPLER	254	-
VACCUM PUMP		16470
PRINTER	4528-T	2128

AR30078

APPENDIX F

ChemWest Analytical Laboratories Quality Assurance Program

# QUALITY ASSURANCE PROGRAM

CHEMIEST Analytical Laboratories, Inc.
6000 North Market Boulevard
Sacramento, CA 95834

8 April 1987

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## INTRODUCTION

The entire CHEMMEST staff is dedicated to providing reliable, high quality analytical data to our clientele. CHEMMEST management believes that Quality Assurance is not just a management function, but that every individual in the laboratory is responsible for ensuring the quality of their analytical data. Therefore, each person within the laboratory is trained in evaluating data, monitoring control limits, and taking the corrective action necessary to assure a reliable, high quality product for all CHEMMEST customers.

CHEMMEST's designated Quality Assurance Officer, Steve Madden, works closely with the Vice President of Technical Services and acting Quality Assurance Director, Dr. Jill Henes, and the various Technical Managers, to assure that all CHEMMEST data is consistently reliable and of the highest quality.

## **QUALITY RSSURANCE PROGRAM**

a Sample Preservation, Receipt, Management, and Tracking

When requested, CHEMUEST will provide our clients with the proper containers for samples, by matrix and method.

Upon receipt, all incoming samples are checked by the Sample Control Department for irregularities and chain of custody discrepancies. All irregularities and discrepancies are noted, and when necessary, the client is immediately notified as part of the corrective action process.

Rfter receipt, samples are logged—in to the CHEMEST system as per the Sample Control SOP. The sample Project Manager is responsible for tracking the samples throughout the laboratory. All samples are maintained in a secure area by the Sample Control Bepartment. During all stages of sample analysis, all sample associated documents are signed and dated by the analysts performing the work. These items are also reviewed, signed, and dated by the individual Laboratory Managers.

e Method Specific Analytical Quality Assurance/Quality Control

Each laboratory (ie. 6C/MS, 6C, Inorganics, etc.) has specific method SOP's that detail the preparation of standards, the documentation of instrument maintenance, the number and type of QC samples, the calibration of instruments, the specific quality control parameters and acceptance criteria, and the corrective action for out of control situations.

After initially demonstrating that the analytical method is in control by means of method specific proficiency or validation testing, the data generated from the analysis of quality control samples, such as matrix spikes and blanks, is evaluated against the applicable quality control acceptance criteria and used to verify that the method is in control. QR/QC summary reports, whether for internal or client requested QR/QC, are generated by the individual laboratory personnel, who check for compliance with QC acceptance criteria. Richerence to the QC acceptance criteria is assured by reviews performed by the individual Laboratory Managers, the Project Manager, the Quality Assurance Officer, and the Vice president of Technical Services.

# o Data Validation and Report Approval Process

After the production of data, both the analyst and the Laboratory Manager review the data for accuracy and completeness. The individual Laboratory Manager is responsible for assuring that all quality control parameters are within the quality acceptance specifications, that all customer required QC requirements are met, and that all calculations are correct as reported. This validation is augmented by the Project Manager, the Quality Assurance Officer, and the Vice President of Technical Services.

The final report is prepared by the Project Manager with the assistance of the Document Control Department, and is subject to approval by the individual Laboratory Managers and the Uice President of Technical Services, who sign the report.

The Document Control Department staff prepares final QR/QC reports, as per the appropriate SOP, with the assistance of the Quality Assurance Officer. They are approved by the Vice President of Technical Services. Quality Assurance/Quality Control records are maintained in the Document Control Department under the direction of the Quality Assurance Officer and the Vice President of Technical Services.

### o Certifications and Performance Evaluations

It is the responsibility of the Quality Assurance Officer and the Vice President of Technical Services to assure that all license and certification requirements are met.

Performance evaluations are conducted under the direction of the Vice President of Technical Services, the Quality Assurance Officer, and the assigned Project Manager. In addition to both State and Federal performance evaluation samples, CHEMUEST analyzes EPR check samples, outside vendor check samples, in house blind check samples, and periodic performance evaluation samples submitted by our sister organization, CompuChem, as part of the corporate Quality Assurance Plan.

# SAMPLE PREPARATION, RECEIPT, MANAGEMENT, AND TRACKING

o Containers, Preservatives, and Storage

Both the type of container, proper preservative, and correct storage conditions for use in the collection of samples for analysis are quite method specific. CHEMUESI utilizes lists of recommended containers, preservatives, and storage conditions, such as in the Federal Register, October, 1984, as well as specific recommendations in individual methods to guide our clients in making the correct choice for particular samples. The details of this selection process are given in the appropriate Sample Control SOP.

o Receipt, Chain of Custody, and Disposal

All incoming samples are checked by the CHEMMEST Sample Control Department for irregularities, such as broken or leaking containers, errors in labeling or descriptions, and chain of custody discrepancies. All irregularities are noted on the chain of custody, and when necessary, the client is immediately notified as part of the corrective action process.

After receipt, samples are logged in to the CHEMEST system, assigned a CHEMEST number, and a sample folder, as per the Sample Control SOP. The signed and dated chain of custody is placed into the sample folder along with any other pertinent traffic information. A Project Manager is assigned and is responsible for tracking the status of the samples throughout the laboratory. Samples are kept in a secure area by the Sample Control Department. Samples are signed out in the Sample Control Department sample log books by those Departments where extraction or analysis will be performed. While residing in those Departments, the samples are kept in secure storage. During all stages of sample analysis, sample log books, laboratory worksheets, workbooks, and/or any other associated documents are signed and dated by the analysts performing the work. These items are also reviewed, signed, and dated by the individual Laboratory Managers.

The samples remain in the custody of the Sample Control Department until they are disposed of. The hold time of the samples before disposal is governed by method, contract, or client requirements. These requirements are detailed in the appropriate Sample Control SOP, the specific contract, or the client folder.

# METHOD SPECIFIC RHOLYTICAL QUALITY ASSURANCE/QUALITY CONTROL

o Sources of Reagents, Chemicals, and Standards

CHEMUEST obtains the reagents, chemicals, and standards used for extraction, calibration, spiking, and reference from a variety of connercial and government sources, in both neat and solution form. Individual laboratory and method SOP's indicate the source of these materials. Lot numbers and other pertinent information about the various materials are documented in the appropriate laboratory log or workbooks. These sources include, but are not limited to, the following list.

- e Aldrich
- o J.T. Baker
- a Banca
- o Cambridgé Isotope Labs
- o Chen Service
- o EM Science
- o fisher
- a Kodak
- o Mallinkredt
- a MSD Isotopes
- o Stohler/KOR
- o Supelco
- o UUR Scientific

# e Preparation and QC of Standards

The preparation and QC of standards for a particular analysis is detailed in the specific method SQP for that analysis. The preparation and QC of all standards share some common conditions, which include, but are not limited to, the following:

## o CC of solvents

- o Water
  - o Use better than ASTM Grade I water
  - o Blanks are analyzed by the techniques used for samples

# o Organics

- o Use residue grade
- o Extraction solvents (ie methylene chloride, hexane) are concentrated and analyzed by the techniques used for samples (ie SC, SC/MS)
  - o Non-extraction solvents are analyzed by the techniques used for samples
- o Preparation of Standards
  - o Use clean, volumetric glassware
  - Use either calibrated analytical balances or volumetric glassware (ie microsyringes) for measuring neat or dilute standards
- o QC of Prepared Standards
  - Initially, new standards are analyzed and compared for traceability to NBS or EPR reference standards, if available.
  - o Repeat standards are enalyzed and compared to the last preparation of the standard.
  - o Traceablility standards are run every six months or when an out of control situation occurs.

## a Instrument Maintenance

o Gas Chromatographic/Mass Spectrometric Systems:

A gas chronatographic/mass spectrometric system (6CHSS) includes:

- o The basic GCMS unit, with all of it's associated GC, MS, pneumatic, and vacuum equipment;
- o The associated computer and it's peripherals:
- o All associated concentration/injection devices.

Each SCMSS is assigned a number or letter designation, and it's own bound, numbered Instrument Maintenance/Repair Log. All maintenance, repairs, or changes performed on a SCMSS, whether done by CHEMMEST staff or an outside vendor, are documented in the associated log book. These events includes

- & Electronic/mechanical maintenance/repair;
- o firmware changes:
- o Software changes:
- o Consumables replacement (ie. traps, columns, etc.).

In addition to the bound log books, there are files within the BCMS laboratory to hold any additional documentation not easily entered into the log books. Examples include:

- o Maintenance/repair receipts:
- o Vendor generated software change documentation:
- o Schematic/diagrams of system changes.

- o Gas Chromatographic Systems:
  - A gas chromatographic system (GCS) includes:
    - o The basic GC oven, with it's associated injectors and pneumatics;
    - o All integral and peripheral detectors;
    - o RIL associated concentration/injection devices.

Each 6CS is assigned a number or letter designation, and it's own bound, numbered Instrument Mointenance/Repair Log. All maintenance, repairs, or changes performed on a 6CS, whether done by CHEMUEST staff or an outside vendor, are documented in the associated log book. These events include:

- o Electronic/mechanical maintenance/repair:
- o firmware changes:
- o Pneumatic changes:
- o Consumables replacement (ie. syringes, traps, but not septa
  = these are documented on cards attached to each
  instrument)

In addition to the bound log books, there are files within the chromatography laboratory to hold any additional documentation not easily entered into the log books. Examples include:

- o Maintenance/repair receipts
- o Vendor generated detector maintenance/repair documentation
- o Schematics/diagrams of system changes

o Chromatography Data Requisition Systems

The chromotography data acquisition system (CDRS) includes:

- o The basic computer(s):
- o All peripherals attached to the main computer, such as disk drives, terminals, A/D's, that are not part of any other system.

The EDRS has it's own bound, numbered Instrument Maintenance/Repair/General Record book. All maintenance, repairs, changes, and pertinent information concerning the CDRS, whether coming from CHEMMEST staff or an outside wendor, is documented in the record book. In addition to the bound record, there are files within the chromatography laboratory to hold any additional information not easily entered into the record book.

o Inductively Coupled Argon Plasma Systems

An inductively coupled argon plasma system (ICRPS) includes:

- o The basic ICRP unit, with all of it's associated pneumatic and vacuum equipment:
- o The associated computer and it's peripherals:
- o All associated concentration/injection devices.

Each ICRPS is assigned a number or letter designation, and it's own bound, numbered Instrument Maintenance/Repair Log. All maintenance, repairs, or changes performed on a ICRPS, whether done by CHEMMEST staff or an outside wendor, are documented in the associated log book. These events includes

- o Electronic/mechanical maintenance/repair:
- o firmware changes:
- o Software changes:
- o Consumables replacement.

In addition to the bound log books, there are files within the Inorganic laboratory to hold any additional documentation not easily entered into the log books. Examples includes

- o Maintenance/repair receipts:
- o Vendor generated software change documentation:
- o Schematic/diagrams of system changes.

## o General Instruments:

For those major instruments not covered specifically (ie IR's, RR's, etc.), each unit is assigned a number or letter designation and it's own bound, numbered Instrument Naintenance/Repair Log. Most smaller instruments are assigned a group Instrument Maintenance/Repair Log. All maintenance, repairs, or changes performed on the instrument(s), whether done by CHEMUEST staff or an outside vendor, are documented in the associated log book. These events includes

- o Electronic/mechanical maintenance/repair:
- o firmware changes:
- o Software changes:
- o Consumables replacement.

In addition to the bound log books, there are files within the specific laboratory to hold any additional documentation not easily entered into the log books. Examples include:

- o Maintenance/repair receipts:
- o Vendor generated software change documentation:
- o Schematic/diagrams of System Changes.

# o Documentation Reviews

Rll maintenance, repair, and general log and record books within the individual laboratories are reviewed by the Laboratory Manager at a frequency no less than every two months to insure that they are being maintained. A spot review of the associated files is also conducted at this same frequency to insure their proper maintenance as well.

# o Analysis of QC Samples

#### c Blanks

Each specific method SOP includes the particular blank requirements for that method. The analysis of blanks includes, but is not limited to, the following general considerations.

- o R minimum of one blank is extracted each time when samples are extracted using a particular extraction technique.
- o The blank associated with a set of extracted samples is analyzed at least once on each instrument used for the analyses of those samples.
- o for non-extracted samples, instrument or system blanks are analyzed at least once during the calibrated analysis period of the instrument or system.
- o For non-extracted samples, instrument or system blanks are analyzed when either carryover contamination is expected to occur, or when contamination is suspected.
- o Reagent blanks are analyzed when changes are made in reagents for a particular method, or when contamination is suspected.

# o Replicates and Spiked Samples

Each specific method SOP includes the requirements for the analysis of replicates, matrix spikes/matrix spike duplicates (MS/MSD), and blank spikes (BLS) for that method. In addition, the particular requirements of the individual client also govern the analysis of replicates and matrix spikes. The analysis of replicates and matrix spikes includes, but is not limited to, the following general considerations.

# o Replicates:

o Replicates are only analyzed when they are specifically requested or required by the individual client or method.

# o Spiked Samples:

- o An MS/MSD pair, and a BLS, are prepared at a -- frequency of 10% of the incoming samples, by method and matrix.
  - o An MS (matrix spike) represents the spiking of a sample with a known amount of target analyte(s).
  - ---- An MSD (matrix spike duplicate) represents a duplicate of the MS, using the same sample.
    - o A BLS (blank spike) represents the spiking of a blank with a known amount of target analyte(s).
- o An MS/MSD pair is analyzed per extraction batch or every 20 samples, per matrix, whichever is more frequent.
  - o If the MS/MSD fails to meet the quality acceptance criteria, the BLS is also analyzed to demonstrate that the system is in control.

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### o Instrument Calibration

### o 6C/MS Systems

The gas chromatographic/mass spectrometry systems are calibrated for mass and then tuned using specific instrument and method parameters. They are then calibrated for quantitation using either the external or the internal standard techniques. Specific methods may impose variations and/or different acceptance criteria upon both the tuning and calibration techniques. These specific requirements are found in the CHETMEST SOP covering the particular method in question.

# o Mass Calibration and Tuning:

Calibrating and tuning the SCMS systems is both instrument and method specific, and includes, but is not limited to, the following general actions:

- o Introduction of the proper mass calibration compound into the BCMS system (ie FC43 [perfluorotributylamine]);
- o Running the proper 60% calibration procedure (ie CR on the Finnigan 5100's):
- o Bocumenting the calibration:
- a Introduction of the proper tuning compound into the GCMS system (ie BF8 Ebronofluorobenzenel for volatiles: BFTPP Edecafluorotriphenylaminel for semi-volatiles):
- o Running the proper 6CMS tuning program (ie. MTUNE on the Finnigan 5188)
- o Making the correct instrument adjustments to meet the method specific tuning criteria;
- s Documenting the tune.

### e External Standard Calibration Procedure:

For each analyte, or group of analytes, five concentration levels of standards are prepared by adding aliquots of one or more stock standards to volumetric flasks and diluting to volume with an appropriate solvent. One of the standards should be at a concentration near the wethout detection limit. The other concentrations should define the working range of the system.

Each of the calibration standards %s introduced into the SCHS system using the technique to be used for introduction of actual samples (ie. 2- to 5-ul liquid injections, purge & trap, etc.). A series of Calibration Factors (CF's) is calculated for each analyte, at each standard concentration, for the mass peak of interest that is specific for that analyte.

## amount introduced

- total response compounds use the total area of all peaks used for quantitation).
- o If the percent relative standard deviation (XRSD) between the Cf's is less than 25% over the working range, linearity through the origin can be assumed and an average CF can be used for quantitation.
- o If this criteria is not met, the standard analyses must, be repeated if quantitation is to be performed.

The working average Calibration Factor must be verified on each working day by the introduction of one or more calibration standards. The frequency of verification is method specific, and varies from once per day to an average of once every five samples.

\_\_\_\_ o If the response of any analyte varies from the predicted response by more than \*/- 20%, a new calibration curve must be prepared for that analyte.

· · · (R1-R2)

o The Percent Difference = ----- x 100, where R1 = R1 the CF from the first analysis, and R2 = the CF from the second analysis.

### o Internal Standard Calibration Procedure:

For each analyte, or group of analytes, five concentration levels of standards are prepared by adding aliquots of one or more stock standards to volumetric flasks. In addition, a known and constant amount of one or more internal standards (IS's) is added to each volumetric flask and they are then diluted to volume with an appropriate solvent. One of the standards should be at a concentration near the method detection limit. The other concentrations should define the working range of the system.

Each of the calibration standards is introduced into the GCMS system using the technique to be used for introduction of actual samples (ie. 2- to 5-ul liquid injections, purge & trap, etc.). A series of Calibration Factors (Cf's) is calculated for each analyte, at each standard concentration, for the mass peak of interest that is specific for that analyte.

o The Calibration Curve is a plot of amount introduced us. the relative response (RR).

o The RR = response of analyte response of IS

# mass introduced

- o The CF = ------------------------ (for multiresponse total relative response compounds use the total area of all peaks used for quantitation).
- o'If the percent relative standard deviation (XRSD) between the CF's is less than 25% over the working range, linearity through the origin can be assumed and an average CF can be used for quantitation.
- o If this criteria is not met, the standard analyses must be repeated if quantitation is to be performed.

The working average Calibration factor must be verified on each working day by the introduction of one or more calibration standards. The frequency of verification is method dependent, and varies from once per day to an average of once every five samples.

- o If the response of any analyte varies from the predicted response by more than \*/- 20%, a new calibration curve must be prepared for that analyte.

### o Chromatography Systems

The chromatographic systems are calibrated using either the external of the internal standard techniques. Specific methods may impose variations and/or different acceptance criteria upon these two techniques. These specific requirements are found in the CHEMUEST SOP covering the particular method in question.

### o External Standard Calibration Procedures

for each analyte, or group of analytes, five concentration levels of standards are prepared by adding aliquots of one or more stock standards to volumetric flasks and diluting to volume with an appropriate solvent. One of the standards should be at a concentration near the method detection limit. The other concentrations should define the working range of the system.

Each of the calibration standards is introduced into the chronatography system using the technique to be used for introduction of actual samples (ie. 2- to 5-ul liquid injections, purge & trap, etc.). Both a Calibration Curve, and a series of Calibration Factors (CF's) at each standard concentration, is calculated for each analyte.

o The Calibration Curve is a plot of amount introduced us. detector response.

### amount introduced

- o The Cf = ------ (for multiresponse total response compounds use the total area of all peaks used for quantitation).
- o If the percent relative standard deviation (XRSD) between the CF's is less than 20% over the working range, linearity through the origin can be assumed and an average CF can be used for quantitation.
- o If the above criteria is not met, the Calibration Curve can be used for quantitation if the residual, r, is greater than 8.995.
- o If neither criteria is met, the standard analyses must be repeated if quantitation is to be performed.
- o If the quantitation criteria are not met, documentation of the ability to see the required 0.805 minimum detectable concentration is sufficient to determine the presence or absence of target compounds.

The working average Calibration Factor or Calibration Curve must be verified on each working day by the introduction of one or more calibration standards. The frequency of verification is detector dependent, and varies from once per day to an average of once every five samples.

- \_\_\_o If the response of any analyte varies from the predicted response by more than +/- 15%, a new calibration curve must be prepared for that analyte.
  - (R1-R2)
    o The Percent Difference = ---- x 100, where R1 =
    R1
    the CF from the first analysis, and R2 = the CF from
    the second analysis.
- o Internal Standard Calibration Procedure:

For each analyte, or group of analytes, five concentration levels of standards are prepared by adding aliquots of one or more stock standards to volumetric flasks. In addition, a known and constant amount of one or more internal standards (IS's) is added to each volumetric flask and they are then diluted to volume with an appropriate solvent. One of the standards should be at a concentration near the method detection limit. The other concentrations should define the working range of the system.

Each of the calibration standards is introduced into the chromatography system using the technique to be used for introduction of actual samples (ie. 2- to 5-ul liquid injections, purge & trap, etc.). Both a Calibration Curve, and a series of Calibration Factors (CF's) at each standard concentration, is calculated for each analyte.

- o The Calibration Curve is a plot of amount introduced us. the relative detector response (RR).
  - o The RR = response of analyte response of IS

### mass introduced

- total relative response compounds use the total area of all peaks used for quantitation).
- o If the percent relative standard deviation (XRSD) between the CF's is less than 20% over the working 80% range, linearity through the origin can be assumed and an average CF can be used for quantitetion.

- o If the above criteria is not met, the Calibration Curve can be used for quantitation if the residual, r, is greater than 0.995.
- o If neither criteria is met, the standard analyses must be repeated if quantitation is to be performed.
- o If the quantitation criteria are not met, documentation of the ability to see the required minimum detectable concentration is sufficient to determine the presence or absence of target compounds.

The working average Calibration Factor or Calibration Curve must be verified on each working day by the introduction of one or more calibration standards. The frequency of verification is detector dependent, and varies from once per day to an average of once every five samples.

o If the response of any analyte varies from the predicted response by more than +/- 15%, a new calibration curve must be prepared for that analyte.

(R1-R2)

o The Percent Difference = ---- x 100, where R1 = R1.

the CF from the first analysis, and R2 \* the CF from the second analysis.

### o ICAP Systems

The inductively coupled argon plasma systems are calibrated daily by an external standard calibration process. Specific methods may impose variations and/or different acceptance criteria upon this calibration. These specific requirements are found in the CHEMMEST SOP covering the particular method in question.

o Daily External Standard Calibration Procedure:

For each analyte, or group of analytes, to be analyzed, initial calibration standards are prepared by adding aliquots of one or more stock standards to volumetric flasks and diluting to volume with an appropriate solvent. These standards should be at concentrations that define the maximum range of the method. Continuing salibration standards, containing the same analyte(s) as the calibration standards, are prepared in the same manner, but at approximately 50% of the calibration standard concentrations.

The appropriate initial calibration standard, followed by an appropriate blank solution, are introduced into the ICRP system in duplicate using the technique usad for the introduction of actual samples. The ICRP system calculates a response factor based on the sytem response to both the standard and blank. This is followed by duplicate introductions of the appropriate continuing calibration standard. The continuing calibration standard is analyzed after every ten samples.

- a Results from the duplicates of each sample or standard must have a percent relative standard deviation (IRSD) less than or equal to 20%.
  - o If this criteria is not met, the sample or standard analysis must be repeated.
- o Results from the continuing calibration standards must fall within +/- 13% of the expected value.
  - o If this criteria is not met, the standard analysis must be repeated.
  - -o If the standard still does not meet the criteria, the entire standardization procedure is repeated.

### o ARS Systems

The atomic absorption systems are calibrated daily by an external standard calibration process. Specific methods may impose variations and/or different acceptance criteria upon this calibration. These specific requirements are found in the CHEMMEST SOP covering the particular method in question.

o Daily External Standard Calibration Procedures

For each analyte, or group of analytes, to be analyzed, initial calibration standards, at three to four different concentrations, are prepared by adding aliquous of one or more stock standards to volumetric flasks and diluting to volume with an appropriate solvent. One of the standards should be at concentrations near the detection limits of the method. Continuing calibration standards, containing the same analyte(s) as the calibration standards, are prepared in the same manner, but at approximately the mid-point of the calibration standard concentration ranges.

Each of the appropriate initial calibration standards, followed by an appropriate blank solution, are introduced into the RR system in replicate using the technique used for the introduction of actual samples. The RP system calculates a set of response factors based on the sytem response to both the standards and blank, and displays the results as a calibration curve. This is followed by replicate introductions of the appropriate continuing calibration standard. The continuing calibration standard is analyzed after every ten samples.

- o Acceptability of system generated calibration curves is made by visual inspection of the curves.
  - o If the curves are judged unacceptable, the calibration standards are reanalyzed.
- o Results from the replicates of each sample or standard must have a percent relative standard deviation (XRSD) less than or equal to 20%.
  - o If this criteria is not met, the sample or standard analysis must be repeated.
- o Results from the continuing calibration standards must fall within +/- 13% of the expected walue.
  - o If this criteria is not met, the standay analysis must be repeated. AR300809
  - o If the standard still does not neet the criteria, the entire standardization procedure is repeated.

# o General Instrument Systems

For general analytical systems and methods, calibration is carried out in accordance with both instrument manufacturer's specifications and the particular requirements of specific analytical methods. These specific requirements, as well as the various acceptance criteria, are found the the CHIMIEST SOP covering the particular method in question.

# e Quality Acceptance Criteria

The CHEMIEST objective for precision and accuracy of analytical data is to use either the Environmental Protection Agency (EPA) criteria for both precision and accuracy of analyses, as listed in some of their published methodology and analytical contracts, or CHEMIEST laboratory generated performance data, to evaluate the quality acceptance limits of the data. The QC criteria for matrix spikes (MS°s), matrix spike duplicates (MSO°s), or blank spikes (BLS°s) are method specific and are detailed in the laboratory SOP for the particular analysis. The acceptable limits for these items includes, but is not limited to, the following general considerations, as appropriate.

### o Precisions

- o The relative percent difference (RPD) criteria, as "published by the EPR in the Statement of Work (SDU) for the Contract Laboratory Program (CLP) for organic and inorganic analysis, and those generated from Laboratory performance data, are used to determine the QC acceptance of the MS/MSD pairs.
- o The RPD criteria for greater then one-helf of the compounds spiked must be met per analysis.
  - o If the criteria is not met, the MS/MSD pair is reanalyzed, and, if necessary, reextracted and reanalyzed.

### o Accuracy:

- o The percent recovery criteria, as published by the EPR in the SOU for the CLP for organic and inorganic analysis, the October 26, 1984 Federal Register, and those generated from laboratory performance data, are used to determine the QC acceptance of MS, MSD, and BLS percent recoveries (IREC).
- o for CLP analyses, the XREC criteria for greater than one-half of the compounds spiked must be met per analysis.
  - o If the criteria is not met, the MS/MSD pair is reanalyzed, and, if necessary, reextracted and reanalyzed.
- o For EPR 600 series method analyses, all of the XREC criteria must be met per analysis.
  - o If the criteria is not met, the BLS is # 183008 | |

- o If the criteria is not met for the BLS, the entire sample batch is reprocessed.
- o for other method analyses, the RPD and XREC criteria are established by the statistical evaluation of 21 data points derived from the specific analysis.
  - o The mean (X) and standard deviation (SD) of the data are calculated, and the control ranges are set at X +/- 2 x SD for a warning limit, and X +/- 3 x SD for an action limit.

# o But of Control Situations

There are a number of conditions that could constitute an "out of control" situation for an instrument, an analysis, or a method.

o Out of Control Situations for Instruments:

For instruments, these are usually mechanical or electronic problems that require either maintenance or repair. In such cases, logical troubleshooting steps, as detailed in specific instrument SOP's or manuals, are undertaken to isolate the problem, and correction is done by either CHEWEST staff or outside vendors trained in instrument maintenance or repair.

o Out of Control Situations for an Analysis:

For a particular analysis, but of control conditions arise when tuning standards, calibration standards, internal standards, or surrogates fail to meet method specific acceptance criteria. Each specific method SOP includes steps to follow for the resolution of these conditions, and they include, but are not limited to, the following general actions, as appropriate.

- o Tuning Standards
  - o Check that all instrument parameters are correct;
  - o Re-analyze at least twice:
  - o Make up new standard solutions;
  - o Re-tune.
- o Calibration Standards/Internal Standards
  - o Check that all instrument parameters are correct;
  - o Reanalyze at least twice;
  - o Perform instrument specific maintenance:
  - o Senerate new calibration data:
  - o Prepare new calibration standards.
- o Surrogates
  - o Check that all instrument parameters are correct:
  - a Check for possible matrix interference with internal standard areas, if applicable;

- o Check for possible matrix interference with surrogate areas:
- o Reanalyze;
  - o Reextract.
- o But of Control Situations for a Method:

For a method, out of control conditions occur when quality control data, such as blanks, duplicates and spikes, do not meet the quality acceptance criteria. Each specific method SOP details the out of control conditions for the method. But of control situations include, but are not limited to, the following general conditions, as appropriate:

### o Blanks

o "Out of control" if background is greater than two to five times the Method Betection Limit or Limit of Betection (method/analyte specific);

## o Duplicates

o "Out of control" if the Relative Percent Difference (RPO) is outside the method specific acceptance criteria:

### o Spikes

- o "Out of control" if the analyte recovery is outside of the method specific acceptance criteria.
- o "Out of control" if the RPD between spike duplicates is outside the method specific acceptance criteria.

Each specific method SOP includes steps to follow for the resolution of these conditions, and they include, but are not limited to, the following general actions, as appropriate.

#### o Blanks

- o Reanaluze:
- o Perform method/instrument specific maintenance:
- o Reanalyze:
- o Reextract.

# o Duplicates

- o Check that no other out of control conditions exist:
- o Reanalyze:
- o Reextract.

# o Spikes

- c Check that no other out of control conditions exist;
- o Reanalyzes
- o Reextract.